

August 18, 1999

MEMORANDUM

SUBJECT: Peer Review of *Guidance on Environmental Data Verification and Validation (EPA QA/G-8)*

FROM: Nancy W. Wentworth, Director /s (Thomas Dixon for)
Quality Assurance Division (8724R)

TO: Peer Review Panel

Thank you for agreeing to review the August 16, 1999 External Review Draft of *Guidance on Environmental Data Verification and Validation (EPA QA/G-8)*, a technical guidance developed by EPA to assist project personnel in documenting the procedures to verify and validate environmental data collected in the field or laboratory. This guidance describes the planning and approach for implementing data verification and validation procedures and the required criteria and information needed to ensure that quality assurance (QA), quality control (QC), and other contractual and technical requirements are met and that the data generated are of known and documented quality. Data verification and validation are an important part of the Quality Assurance Project Plan, a mandatory planning document that describes the necessary QA and QC elements to ensure that collected data will achieve its planned performance criteria.

Your general charge is to review this document to determine its utility and credibility as guidance for planning data verification and validation procedures within projects to be performed under the sponsorship or on behalf of the EPA. We are most interested in a conceptual review, rather than a detailed editorial review. We would like you to address the following considerations as part of your review:

1. Does the document begin with a clear indication of what it aims to address and how it would benefit the reader?
2. Are the definitions for verification, validation, data verification, and data validation presented in Section 1.2 complete and acceptable?
3. Is the stated purpose of data verification and data validation presented in Section 1.4 sufficient?

4. Do you agree with the approach to defining and presenting data verification within the context of the four elements (compliance, correctness, consistency, and completeness) presented in Section 2.2.1? If not, how would you change this approach? Are the elements defined sufficiently, and do they cover all aspects of data verification?
5. Are Tables 2-1 through 2-4, detailing example procedures performed within the four elements of data verification, useful to the reader? If not, how can such a presentation be improved?
6. Do you agree with the two-staged process used to define data validation in Section 2.2.2? If not, how would you change this approach? Are the stages defined sufficiently?
7. Do we sufficiently provide alternative approaches to data validation and options for verifying that the validated data satisfy relevant claims about their authenticity and quality?
8. The implementation of data verification and validation, presented in Chapter 3, focuses primarily on reporting issues. Should we detail other important elements within these implementation presentations?
9. Are you aware of additional references (e.g., automated data verification software) that have been omitted?
10. Are the case studies presented in the appendix useful in observing how data verification and validation are implemented?
11. Is the relationship between data verification and data validation and development of the Quality Assurance Project Plan clearly presented?
12. Overall, how useful will the guidance be to environmental scientists and engineers?

Please feel free to offer comments and suggestions that go beyond this charge, as you see fit.

To meet EPA's schedule for publishing this document, it is important for us to receive your comments by September 15, 1999. Please send written comments to:

Esperanza P. Renard
Quality Assurance Division (MS-104)
U.S. Environmental Protection Agency
2890 Woodbridge Avenue
Edison, New Jersey 08837
Phone: (732) 321-4355
Fax: (732) 321-6640
e-mail: esperanza.renard@epa.gov

Thank you for your time and efforts. I look forward to your contribution.

Attachment

**GUIDANCE ON
ENVIRONMENTAL DATA VERIFICATION
AND VALIDATION**

EPA QA/G-8

United States Environmental Protection Agency
Quality Assurance Division

Washington, DC 20460

PEER REVIEW DRAFT

AUGUST 1999

DO NOT QUOTE OR CITE

FOREWORD

The U.S. Environmental Protection Agency (EPA) has developed this document, entitled *Guidance on Environmental Data Verification and Validation (EPA QA/G-8)*, to provide a standardized working tool for project managers, environmental scientists, and engineers involved in collecting and/or measuring environmental data. Data verification and validation are performed within an environmental project to ensure that the data collection process has produced credible and cost-effective data of known and defensible quality. This document is intended to define the concepts of data verification and data validation of environmental data collected in the field or laboratory, to distinguish between these two processes, to describe the planning and approach for implementing data verification and validation, and to document examples of data verification and validation procedures. Data verification and validation are important elements of a project's Quality Assurance Project Plan (QAPP), a mandatory planning document that describes the necessary quality assurance and quality control elements to ensure that the data collected achieve acceptable performance criteria.

This document is one of a series of guidance documents prepared within the EPA Quality System Series. This series describes the EPA policies and procedures for planning, implementing, and assessing the effectiveness of the Quality System. Questions regarding this document or any other Quality System Series document may be directed to:

U.S. Environmental Protection Agency
Quality Assurance Division (8724R)
Office of Research and Development
401 M Street, SW
Washington, DC 20460
Phone: (202) 564-6830
Fax: (202) 565-2441
e-mail: ord-qad@epa.gov

These documents are available on the EPA Quality Assurance Division website at:

<http://es.epa.gov/ncerqa/qa/index.html>

or by contacting EPA's Quality Assurance Division.

(This page left blank intentionally.)

TABLE OF CONTENTS

	Page
Foreward	ii
List of Acronyms	v
CHAPTER 1 INTRODUCTION	1
1.1 Overview	1
1.2 Definitions	1
1.3 The EPA Quality System	2
1.4 Purpose of Data Verification and Validation	4
1.5 Scope and Intended Audience	4
1.6 Organization of This Document	8
CHAPTER 2 PLANNING THE DATA VERIFICATION AND DATA VALIDATION PROCESSES	9
2.1 Mandate for Performing Data Verification and Validation	9
2.2 What Distinguishes Data Verification from Data Validation?	10
2.3 Case Study for Methods Illustration: Sampling from Emissions of Coal-fired Power Plant	13
CHAPTER 3 IMPLEMENTING THE DATA VERIFICATION PROCESS	15
3.1 The Four Elements of Data Verification, with Example Procedures	15
3.1.1 Compliance	21
3.1.2 Correctness	23
3.1.3 Consistency	24
3.1.4 Completeness	25
3.2 Verifying that MQOs Have Been Met	27
3.3 Reporting the Results of Data Verification	28
3.4 Automated Data Verification	33
CHAPTER 4 IMPLEMENTING THE DATA VALIDATION PROCESS	35
4.1 Reporting the Results of Data Validation	38
REFERENCES	40
APPENDIX A GLOSSARY OF TERMS	A-1
APPENDIX B DESCRIPTION OF RESPONSIBILITIES FOR JOBS DETAILED IN TABLES 3-1 THROUGH 3-4	B-1
APPENDIX C ADDITIONAL CASE STUDIES	C-1

		<u>Page</u>
C.1	Characterizing Human Exposure to Metals	C-1
C.2	Toxicity Testing of POTW Effluent Using <i>Mysidopsis Bahia</i>	C-11

APPENDIX D ISSUES CONCERNING THE VALIDATION OF DATA VERIFICATION SOFTWARE D-1

LIST OF TABLES

		<u>Page</u>
Table 3-1.	Examples of Data Compliance Procedures and Their Purpose	16
Table 3-2.	Examples of Data Correctness Procedures and Their Purpose	18
Table 3-3.	Examples of Data Consistency Procedures and Their Purpose	19
Table 3-4.	Examples of Data Completeness Procedures and Their Purpose	19
Table 3-5.	Example Checklist Entries for Compliance Procedures, As May Occur Within the Three Case Studies	29

LIST OF FIGURES

		<u>Page</u>
Figure 1-1.	Components of the EPA Quality System	5
Figure 1-2.	Project Phase of the EPA Quality System, with Detail Given on the Assessment Phase	6
Figure 1-3.	Life Cycle of an Environmental Data Collection Project	7
Figure 2-1.	The Four Groups (Group A through Group D) of Elements Within a Quality Assurance Project Plan (QAPP), and Those Elements Within Group D Addressing Data Verification and Validation	10
Figure 2-2.	Flow Diagram of the Data Verification and Data Validation Processes	12

List of Acronyms

1		
2	CCB	Continuing Calibration Blank
3	CCV	Continuing Calibration Verification
4	CFR	Code of Federal Regulations
5	COC	Chain of Custody
6	DL	Detection Limit
7	DQA	Data Quality Assessment
8	DQOs	Data Quality Objectives
9	DVV	Data Verification and Validation
10	EPA	Environmental Protection Agency
11	ICB	Initial Calibration Blank
12	ICV	Initial Calibration Verification
13	ICS	Interference Check Sample
14	ISO	International Organization for Standardization
15	LCS	Laboratory Control Sample
16	MDL	Method Detection Limit
17	MQOs	Measurement Quality Objectives
18	NIST	National Institute of Standards and Technology
19	PB	Preparation Blank
20	QA	Quality Assurance
21	QAPP	Quality Assurance Project Plan
22	QC	Quality Control
23	RPD	Relative Percent Difference
24	SOP	Standard Operating Procedure
25	SRM	Standard Reference Material
26	TSA	Technical Systems Audit

CHAPTER 1

INTRODUCTION

1.1 OVERVIEW

This document, *Guidance on Environmental Data Verification and Validation (EPA QA/G-8)*, is part of a series of documents prepared by the Quality Assurance Division of the U.S. Environmental Protection Agency (EPA) to provide guidance and requirements for planning and implementing environmental studies and monitoring operations. This series supports the implementation of EPA's Quality System, which provides the Agency with a framework for ensuring quality in all aspects of an environmental data collection operation.

This document provides guidance for verifying and validating data collected as part of an environmental operation. Specifically, this document defines data verification and validation, identifies a series of verification and validation procedures, and discusses how the elements of data verification and validation are integrated in the planning and implementation of environmental studies.

1.2 DEFINITIONS

For the purposes of consistency, the following definitions will be used throughout this document. These definitions do not constitute the Agency's official use of terms for regulatory purposes and should not be construed to alter or supplant other terms in use. The definitions of other terms used in this document are provided in the glossary in Appendix A.

Verification: The confirmation by examination and provision of objective evidence that *specified requirements have been fulfilled*. In design and development, verification concerns the process of examining a *result of a given activity* to determine *conformance to the stated requirements for that activity*.

Validation: The confirmation by examination and provision of objective evidence that the *particular requirements for a specific intended use have been fulfilled*. In design and development, validation concerns the process of examining a *product or result* to determine *conformance to user needs*.

Data Verification: A consistent, systematic process that determines whether the data have been collected in accordance to the specification of the Quality Assurance Project Plan with respect to compliance, correctness, consistency, and completeness as compared to a standard or contract.

Data Validation: An evaluation of the technical usability of the verified data with respect to the planned objectives or intention of the project. In addition, data validation can

provide a level of overall confidence in the reporting of the data based on the methods used.

Data Quality Objectives (DQOs): Qualitative and quantitative statements regarding the design and management of the effort to support appropriate collection and use of data. DQOs define the data to be collected, determine the most appropriate condition from which to collect the data, and specify the criteria which define the quality and quantity of the data to be collected.

Measurement Quality Objectives (MQOs): Specific goals that clearly describe the performance requirements for a measurement system. MQOs specify acceptance criteria for Data Quality Indicators, such as selectivity, sensitivity, detection limits, bias, precision, representativeness, comparability, and completeness for the collected data.

Quality Assurance Project Plan (QAPP): A document that describes how quality assurance (QA) and quality control (QC) are applied during the life cycle of an environmental data operation to ensure that the results obtained are of the type and quality needed and expected. A QAPP, which also includes a statement of the operation's Data Quality Objectives, is the critical planning document for any environmental data collection operation.

Data Quality Assessment (DQA): A scientific and statistical evaluation of the data to determine whether the verified and validated environmental data are of the right type, quality, and quantity to support their intended use.

1.3 THE EPA QUALITY SYSTEM

A quality system is a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products, and services. A quality system provides the framework for planning, implementing, and assessing work performed by the organization for carrying out QA and QC activities. It is required by all organizations performing work for EPA in order to assure that:

data collected for the characterization of environmental processes and conditions are of the appropriate type and quality for their intended use; and

environmental technologies are designed, constructed, and operated according to defined expectations.

EPA's Quality System is the means by which the Agency implements its quality management process. The Quality System encompasses a variety of technical and administrative elements such as:

- organizational structure;
- policies and procedures;
- responsibilities;
- authorities;
- resources;
- requirements documents; and
- guidance documents.

The Quality System applies to management systems and to the collection, evaluation, and use of environmental data. Also, the Quality System applies to the design, construction, and operation of environmental technology.

The EPA Quality System operates under the authority of Order 5360.1 CHG 1, *Policy and Program Requirements for the Mandatory Agency-wide Quality System* (July 1998). The Order requires all environmental programs conducted by, or on behalf of, EPA to be supported by a mandatory Quality System. As the standard for developing and operating quality systems, EPA has adopted the American National Standard ANSI/ASQC E4-1994, *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*.

Figure 1-1 illustrates the three levels of the EPA Quality System. At the Policy Level, the specifications and guidelines of the ANSI/ASQC E4 as well as elements from the International Organization for Standardization (ISO) 9000 series of quality management standards are combined with Agency policies to provide a framework for the EPA Quality Manual. The Organizational/Program Level is comprised of components of the Quality System that affect management policies and processes that have a broad application to the organization. The Project Level is comprised of the components that are applied to specific projects or programs within an organization that require the collection or generation of environmental data. Documents from the EPA Quality System series that provide details for the given components are given in parentheses in Figure 1-1.

The Project Level of the Quality System is divided into three stages: planning, implementation, and assessment. Establishment of data quality objectives (DQOs) and development of a Quality Assurance Project Plan (QAPP) are the focus of the planning phase. The implementation phase addresses actual data collection, using the methods specified in the planning stage. Data verification and validation are performed within the assessment stage, where analysts use technical knowledge and statistical methods to determine whether or not the data met the user's needs.

Figure 1-2 provides a more detailed illustration of the assessment stage of the Project Level of EPA's Quality System. This figure shows that the assessment begins with verification and validation of the environmental data that were obtained in the implementation stage. Once the individual data points have been verified and validated, Data Quality Assessment (DQA) is

performed using the entire body of data to determine whether the data have met the user's performance criteria as specified in the DQOs for the project or program.

Figure 1-3 illustrates where data verification and validation fall within the life cycle of an environmental project. This figure shows that the inputs to data verification include the data that were obtained from environmental sampling, as well as the data that were obtained from quality assurance (QA) and quality control (QC) samples. Data verification examines these data individually to determine whether the Measurement Quality Objectives (MQOs) that were defined during project planning have been met. Data validation takes the verified data and determines whether the specified MQOs were adequate for the environmental project. Data Quality Assessment is performed on a data set that has been verified and validated.

1.4 PURPOSE OF DATA VERIFICATION AND VALIDATION

The purpose of data verification is to evaluate the extent to which the sample collection and analytical procedures that were prescribed in the QAPP and the contract authorizing the program's execution were followed. The focus is on identifying whether all requirements specified in either the QAPP or the contract have been met, and if not, determining the extent to which requirements failed to be achieved. Example objectives of data verification include:

- ensuring the integrity and stability of samples throughout the project's life cycle,
- evaluating and maintaining instrument performance during sample analysis, and
- ensuring that data values are reported accurately.

The purpose of data validation is to ensure that the measurement system (field and laboratory) meets the users' needs. Example objectives of data validation include:

- ensuring the proper identification and quantification of analytes
- determining the overall usability of the data relative to project objectives.

1.5 SCOPE AND INTENDED AUDIENCE

Objectives of the EPA QA/G-8 document are:

- to define and distinguish between data verification and data validation, as considered within the EPA Quality System
- to identify the requirements and elements of data verification and validation, as well as procedures for performing data verification and validation, to be included within the verification and validation portions of the QAPP, and
- to provide the reader with examples (case studies) of how data verification and validation procedures are established and implemented.

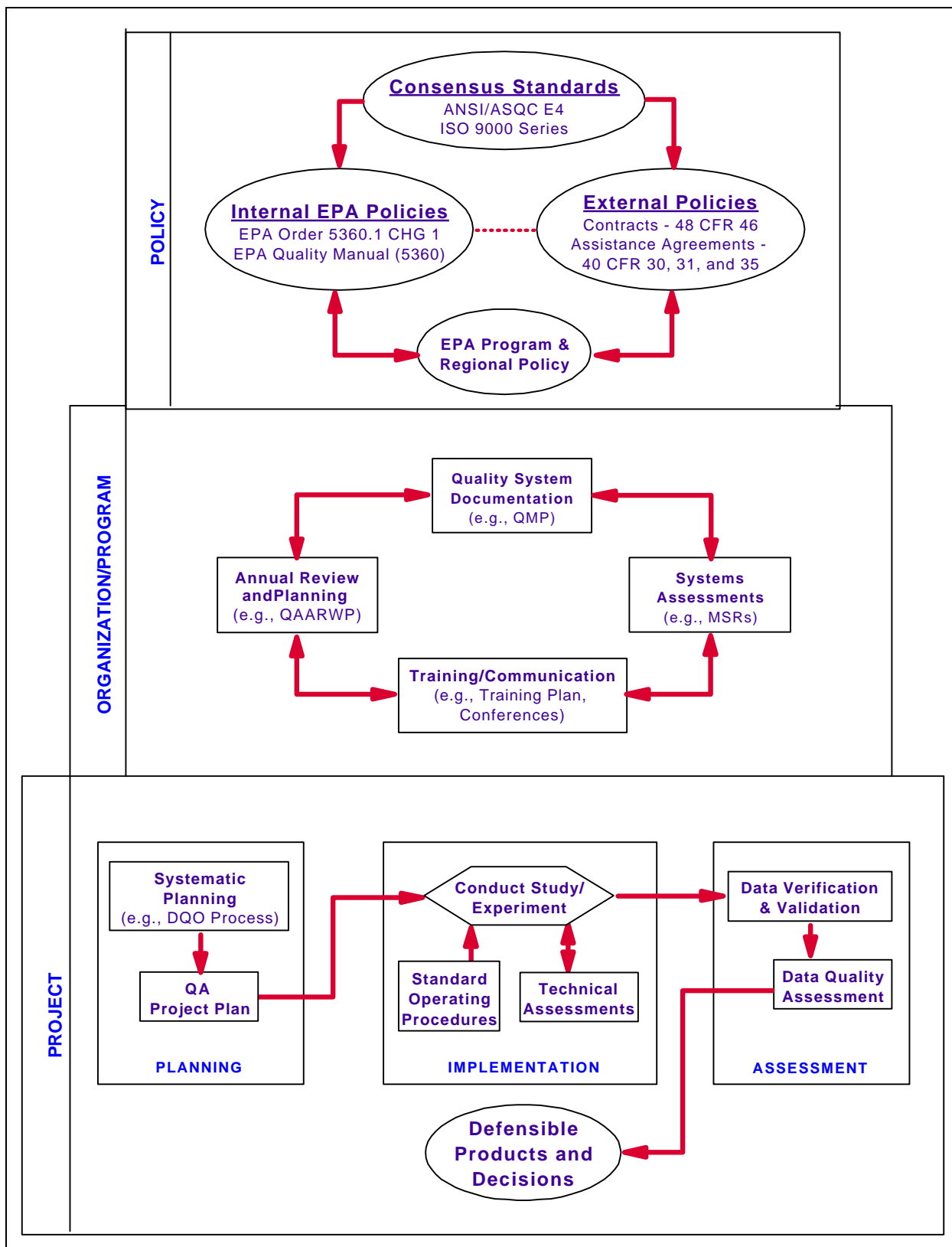


Figure 1-1. Components of the EPA Quality System

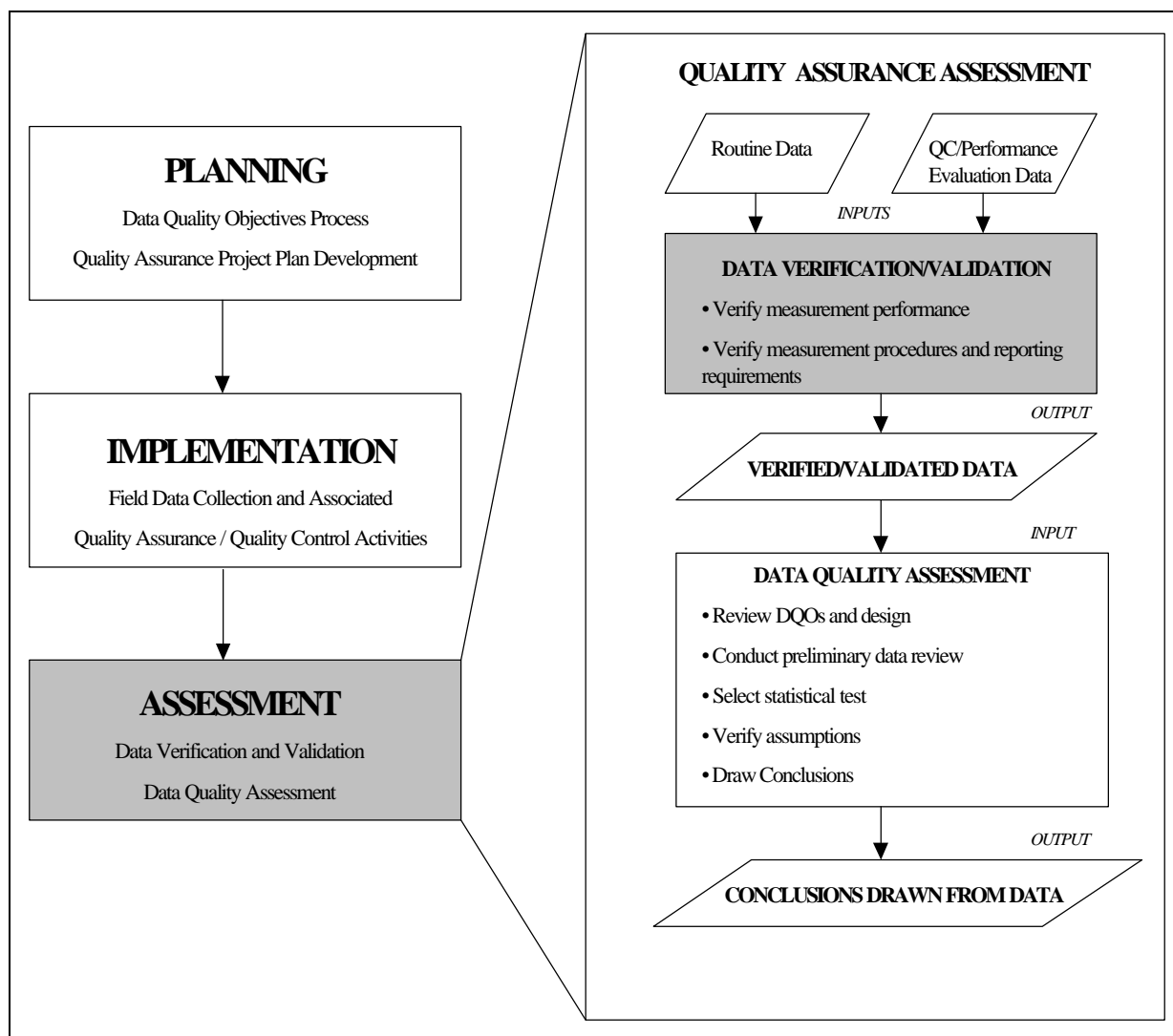


Figure 1-2. Project Phase of the EPA Quality System, with Detail Given on the Assessment Phase

The purpose of this document is to assist managers and planners in planning the approach and methods and/or procedures for verifying and validating environmental data, determining why such procedures are important, and deciding when they should be implemented in an environmental data collection and reporting process. These issues must be considered when preparing the QAPP. Furthermore, this document provides guidance in implementing the data verification and validation procedures that are described in the QAPP. This guidance is relevant to both laboratory and field monitoring and to regulatory policies.

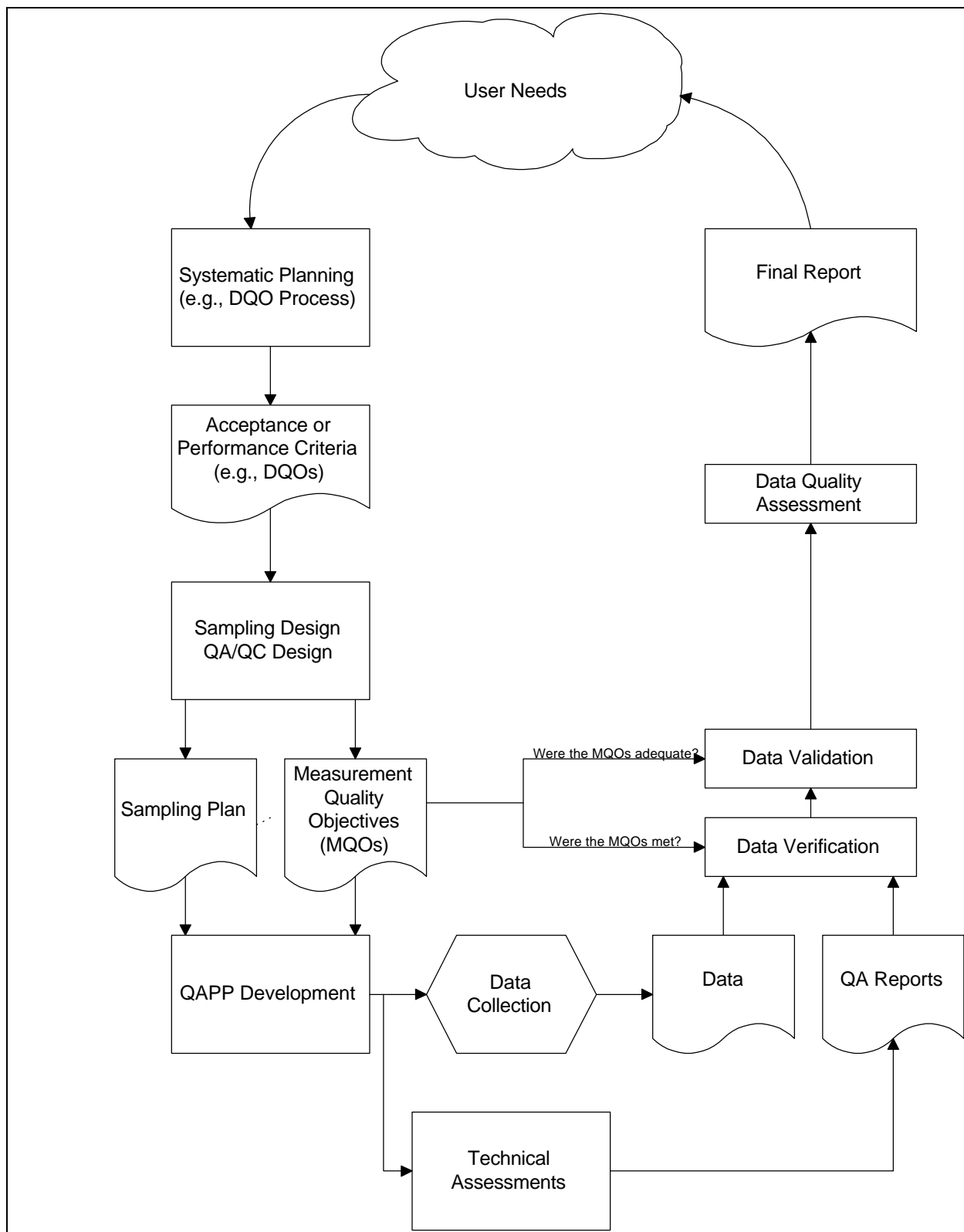


Figure 1-3. Life Cycle of an Environmental Data Collection Project

1 Because the specific approach to performing data verification and validation approach will
2 vary from one project to another according to the project's underlying objectives and approaches
3 taken, there is no single verification or validation plan that will be exactly the same for all such
4 data operations. Therefore, this document does not prescribe a single verification or validation
5 plan. Rather, it provides information about a number of procedures and methods that can be
6 used. The manager of an environmental data collection effort (denoted by "Project Manager" in
7 this document) is required to select data verification and validation methods that are appropriate
8 for the given effort and to present these methods in a QAPP.

9 As this document is meant to be used as a supplement to the document, *EPA Guidance*
10 *for Quality Assurance Project Plans (EPA QA/G-5)*, and to the document, *EPA Requirements for*
11 *Quality Assurance Project Plans (EPA QA/R-5)*, the intended audience for this guidance is the
12 same. In particular, this document has been written for project managers and environmental
13 investigators who will be contributing to a QAPP that will address efforts to collect or generate
14 environmental data in research or technical assessment in the laboratory or in the field.

15 **1.6 ORGANIZATION OF THIS DOCUMENT**

16 The document is divided into the following five chapters:

- 17 • Chapter 1 provides an introduction.
- 18 • Chapter 2 focuses on the planning for data verification and validation, providing
19 more detailed definitions and how these two processes are distinguished.
- 20 • Chapter 3 discusses how data verification procedures are identified and
21 implemented and how the results are used in a typical field monitoring project.
- 22 • Chapter 4 presents guidance on how data validation procedures are identified and
23 implemented.
- 24 • Chapter 5 contains selected references.

25 The appendices support the information supplied in these chapters.

CHAPTER 2

PLANNING THE DATA VERIFICATION AND DATA VALIDATION PROCESSES

While data verification and validation procedures are implemented within the final (assessment) phase of the data life cycle, identifying and planning for these procedures occurs within the first (planning) phase of the data life cycle, as part of quality assurance project plan (QAPP) development. A final verified, validated database can be successfully obtained for data quality assessment only if all necessary and appropriate data verification and validation procedures are identified and included within the project's QAPP, before any data are collected.

This chapter presents additional guidance for the environmental scientist or engineer on what constitutes data verification and data validation and how these two processes differ. As the specific data verification and validation procedures to be applied to a given project are to be documented within the project's QAPP, this guidance supplements the information provided in EPA QA/G-5 and EPA QA/R-5, which provide Agency guidelines and requirements for preparing a QAPP.

2.1 MANDATE FOR PERFORMING DATA VERIFICATION AND VALIDATION

As discussed in EPA QA/G-5 and illustrated in Figure 2-1, the elements of a QAPP are categorized into four "groups" labeled Groups A through D. One of these groups (Group D) is labeled "Data Validation and Usability" and consists of elements that are implemented after the data collection phase of the project has been completed. Two of the QAPP elements within this group specifically concern data verification and validation: "Data Review, Validation, and Verification Requirements" and "Validation and Verification Methods." Therefore, the requirement of performing appropriate data verification and validation procedures on a project is established by requiring the project's QAPP to include these two elements.

Every environmental data collection operation under the auspices of the EPA must follow an Agency-approved QAPP. Furthermore, referring to the elements of a QAPP, EPA QA/R-5 states the following: "All applicable elements defined by the EPA organization sponsoring the work must be addressed in the QAPP. If an element is not applicable, state this in the QAPP." Therefore, because data verification and validation methods and requirements have been identified as elements within a QAPP, the approach to performing data verification and validation procedures must be addressed within the QAPP, or reason(s) that such procedures are not applicable to the project must be given. At a minimum, the plan specified in the QAPP for executing data verification and validation procedures should list these procedures and provide information on how the results of implementing these procedures will be reported.

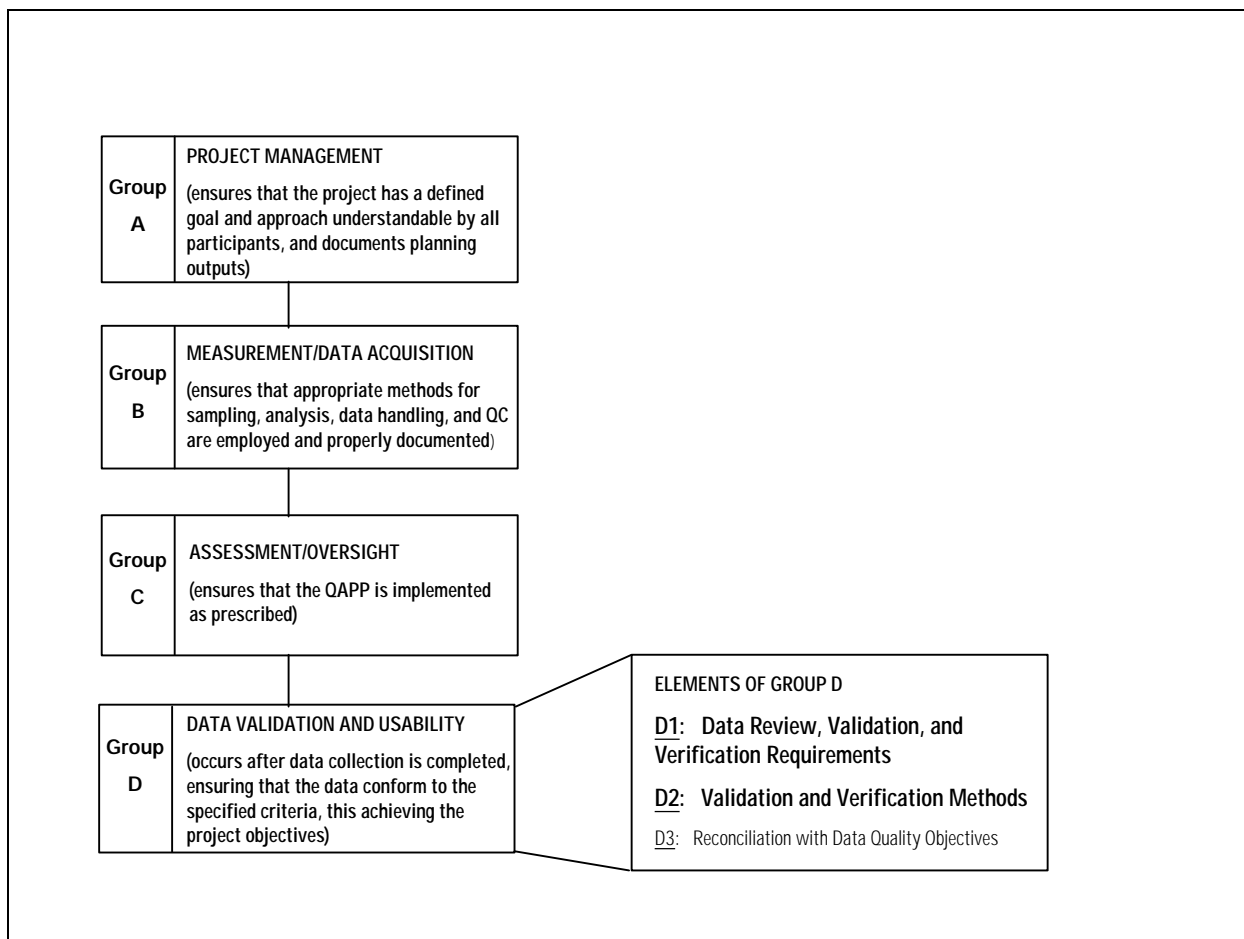


Figure 2-1. The Four Groups (Group A through Group D) of Elements Within a Quality Assurance Project Plan (QAPP), and Those Elements Within Group D Addressing Data Verification and Validation (Sources: EPA QA/G-5, EPA QA/R-5)

2.2 WHAT DISTINGUISHES DATA VERIFICATION FROM DATA VALIDATION?

The similar definitions provided in Chapter 1 for data verification and data validation have frequently caused confusion on determining which responsibilities constitute data verification and which constitute data validation. As a result, different people can have different understandings of these two processes and how to distinguish between them. This section helps clarify the two processes and, in turn, provides additional detail on their definitions and scope.

The key underlying concepts of data verification and data validation are the following:

- Data verification is a systematic, mechanical determination of whether the collected data adhere to pre-defined requirements (e.g., SOPs, MQOs, contractual requirements) detailed in the QAPP and other documents that provide project direction.

- Data validation, performed at a higher level than data verification, is a scientific evaluation of the technical usability of the data in the context of project objectives and the situation in which the project was conducted.

Therefore, data verification focuses on QAPP/contract compliance, while data validation considers technical reliability relative to decision making and meeting project objectives. As a result, data verification is conducted before data validation and all references in this guidance to “data verification and validation” purposely specify verification before validation.

The following example clearly delineates between data verification and data validation. Suppose analyte concentrations were being measured by an instrument having a detection limit of 5 ppb. First, data verification was performed to ensure that the reported data met all necessary criteria defined within the QAPP, including criteria on bias and precision. The data verification process concluded that the reported data had high levels of accuracy and meet all contractual requirements and therefore could proceed to data validation. However, despite the positive outcome of the data verification, the data needed to be used to make decisions at levels around 1 ppb, which is below the method detection limit. Thus, as the data could not be used to meet program objectives and to make necessary decisions, it failed the data validation process.

Figure 2-2 shows the general flow of data through the verification and validation processes. Recall from Chapter 1 that the activities within Figure 2-2 occur in the assessment phase of the project life cycle, and the outcome of these activities is a data set that can be used as input to the Data Quality Assessment (EPA QA/G-9).

The data verification process begins after data have been collected. This is an objective, mechanical process performed on the individual data points and driven by the specifications established in the QAPP. Data verification focuses on whether the data have been collected in accordance with the QAPP specifications and meet compliance requirements as specified by a standard or contract. As indicated in Figure 2-2 and elaborated upon in Chapter 3, the data verification process consists of four procedural elements (Karnofsky, 1997):

Compliance: The extent that adherence to SOPs, QAPP, and/or contractual requirements were followed, achieved, and/or completed successfully, and that conditions under which the data were recorded also met the requirements. Compliance ensures that the data pass numerical quality control tests, including criteria on precision and accuracy, based on parameters or specified limits specified in relevant SOPs and or QAPP.

Correctness: A mechanical, objective check that data collection plans and protocols have been followed and that basic operations and calculations were performed using properly-documented and properly-applied algorithms. Correctness ensures that the reported values are based on properly documented algorithms.

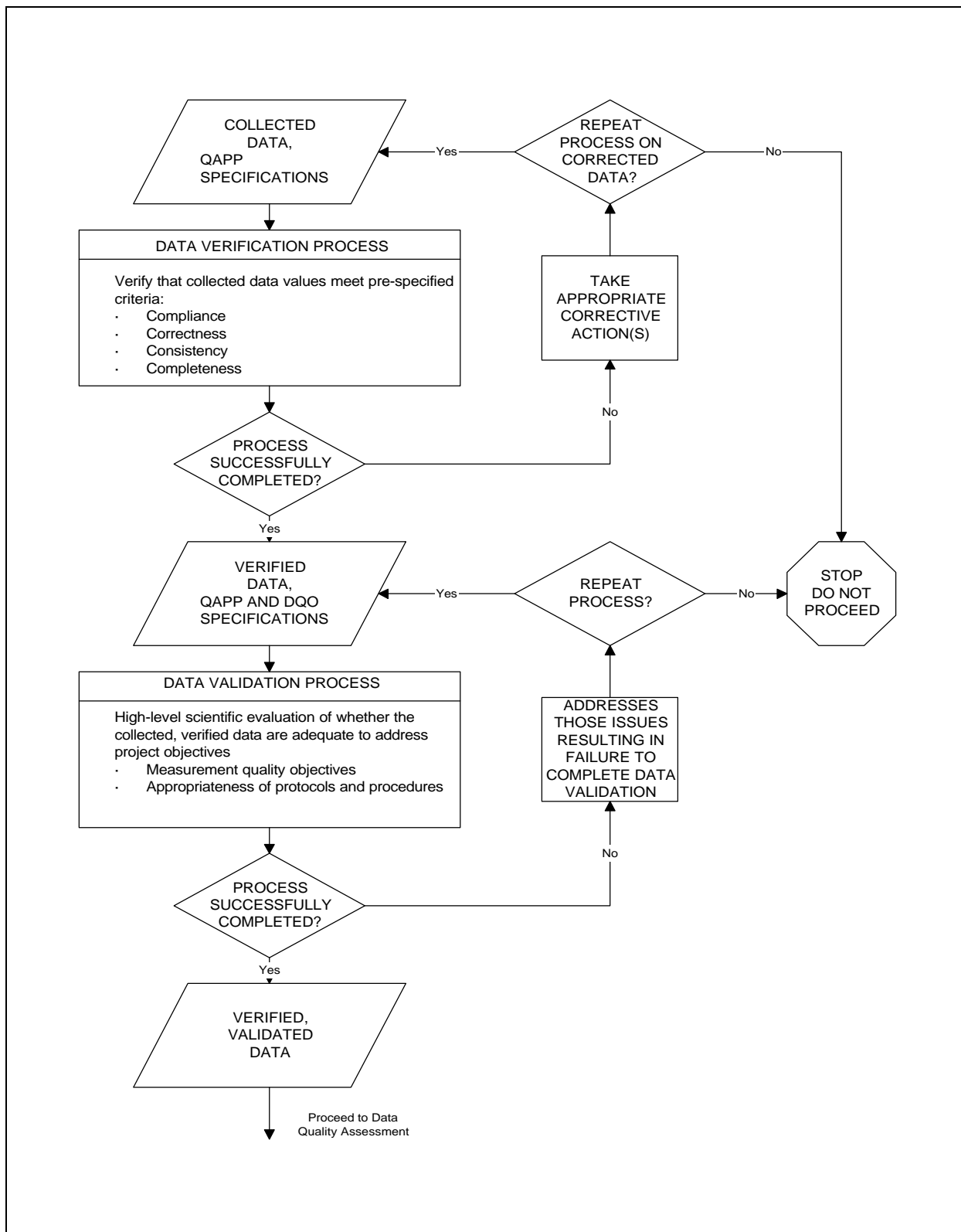


Figure 2-2. Flow Diagram of the Data Verification and Data Validation Processes

1 Consistency: The extent to which data collection procedures were done in a similar
2 manner across different sites (if applicable) and data reporting was done in a similar
3 manner in multiple places. Consistency (also known as comparability) ensures that the
4 reported values are the same when used throughout the project.

5 Completeness: The extent to which all data necessary to perform validation analysis were
6 actually collected. Completeness ensures that a sufficient amount of data and information
7 (relative to the prescribed DQOs) are present.

8 Once data have been successfully verified, a data validation process is performed to
9 evaluate whether the measurement system (field and laboratory) meets the project objectives.
10 Data validation also evaluates the applicability of certain data requirements in the context of the
11 completed project, such as the MQOs. This process is detailed in Chapter 4.

12 Note from Figure 2-2 that certain components of the data verification and data validation
13 processes may be iterated multiple times before achieving data sets that can successfully proceed
14 to the next level. In the data verification process, this may involve making necessary corrections
15 to data values or re-examining certain data verification criteria before repeating the process.

Data Quality Objectives: The overall objective of the project is to conduct a comprehensive assessment of toxic emissions from two coal-fired electric utility power plants as part of an air toxics assessment of this source category. Specific objectives of the project are to:

- achieve at least 85 percent completeness in obtaining the planned data, in order to determine emission levels of the target air toxics in gas, solid, and liquid streams;
- collect sufficient emission and mass loading data to permit calculation of removal efficiencies of pollution control systems and mass balances for the two power plants;
- collect a sufficient quantity of size-fractionated particulate flue gas emissions to permit evaluation of concentration of air toxic emissions as a function of particle size; collect a sufficient quantity of gas sample to establish comparable data for the particulate and vapor phases of air toxic emissions;
- establish comparable data for the hot and stack dilution sampling such that an overall stack target emission accuracy of three is achieved for non-reactive species.

Power plant sampling collects representative samples of gas, solid, and liquid emissions from the power plants for subsequent laboratory analysis. Field sampling data are gathered for various categories, including flue gas sampling data, solid and liquid process sampling data, and operating process data.

Sampling occurs in three test periods, each of two days' duration. The same measurements are made in each of the three test periods (i.e., the three test periods are replicates of each other). Collecting process samples is identical on all six sampling days. However, the two days within each test period are be used for different sets of flue gas measurements. Efforts on one day focus on inorganic measurements, while the other day focuses on organic measurements. Field quality control samples are collected at the specified frequency and acceptance limits prior to transportation to the laboratory.

CHAPTER 3

IMPLEMENTING THE DATA VERIFICATION PROCESS

The data verification process consists of the following:

- Verifies that the values of individual data points meet the criteria specified in the QAPP (e.g., MQOs), and that data collection adheres to SOPs and contractual requirements (compliance).
- Evaluates, in an objective and mechanical manner, whether data collection plans, protocols, and instructions were followed and basic operations and calculations were performed correctly (correctness).
- Evaluates the comparability of data reported over multiple places and collection sites, determining whether collection procedures were followed in the same manner (consistency).
- Obtains all required data and reports all deficiencies (completeness).
- Ensures that the resulting data set is of the form that will facilitate executing the data validation process.

3.1 THE FOUR ELEMENTS OF DATA VERIFICATION, WITH EXAMPLE PROCEDURES

As introduced and defined in Section 2.2, the data verification process consists of four elements: compliance, correctness, consistency, and completeness. For each of these four elements, Tables 3-1 through 3-4 outline procedures or specific steps of procedures that address the goals of each element. The tables include the type of procedure, a description of the procedure, and the job title of one or more individuals generally responsible for performing the procedure. These job titles are not universal, and a single individual may perform the roles of several of the job titles that are shown in the table. Appendix B contains a brief description of the responsibilities associated with each of the job titles listed in these tables.

It should be noted that the procedures in Tables 3-1 through 3-4 are examples of what may be performed within each element of the data verification process. These procedures are not mandatory for every project. Some procedures will be appropriate for some activities and not for others. Also, these lists of procedures may not be exhaustive. It is the responsibility of the project manager to determine which procedures should be implemented and whether other verification procedures are appropriate.

Most, if not all, laboratories have quality assurance (QA) and quality control (QC) plans for sample handling and analysis. Most of these plans include many of the procedures discussed in Tables 3-1.

Table 3-1. Examples of Data Compliance Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
Staff Training and Certification	Verifies that project staff is qualified to perform the work	Project Manager
Sample Custodian Assignment	Reviews the responsibilities of sample custodians in the field and laboratories	
Field Data Collection Audit	Verifies that proper sampling protocols were performed in field sample collection	Field Team Leader
Field Blank and Duplicate Sample Collection	Verifies that the required number of field blanks and field sample duplicates were collected	Field Technical Personnel
Calibration	Confirms that the instruments have been properly calibrated with NIST traceable standard from a source independent of calibration standards (e.g., SRM)	Laboratory Technical Personnel
Interference Check Sample (ICS) Analysis	Verifies the laboratory's inter-element and background correction	
Initial Calibration Blank (ICB) Analysis	Verifies that calibration blank sample was analyzed after calibration standards, and the analysis results were within acceptable limits	
Initial Calibration Verification (ICV) Analysis	Demonstrates that the initial calibration was valid by analyzing a mid-range standard made from a source independent of the working standards	
Continuing Calibration Verification (CCV) Analysis	Checks the continued validity of the initial calibration	
Continuing Calibration Blank (CCB) Analysis	Verifies that the CCB was analyzed at the proper frequency and location during the run	
Method Blank Collection and Analysis	Verifies that at least one preparation blank (PB) was processed through sample preparation and analysis	
Blank Sample Analysis	Analyzes blank samples to measure background contamination, with different types of blanks measuring different types of bias and contamination: 1. Field blank (transport and field handling bias) 2. Reagent blank (contaminated reagent) 3. Rinsate blank (contaminated equipment) 4. Method blank (response of entire laboratory analytical system)	
Calibration Check Sample Analysis	Performs the following checks on the proper calibration of instruments: 5. Zero check (calibration drift) 6. Span check (calibration drift and memory effects) 7. Mid-range check (calibration drift and memory effects)	

Table 3-1. Examples of Data Compliance Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
Analysis of Duplicate Samples	<p>Demonstrates acceptable system precision using the following methods:</p> <ol style="list-style-type: none"> 8. Generate data to determine long-term precision of the analytical method on various matrices. 9. Calculate Relative Percent Difference (RPD) for original and duplicate samples greater than 5 times the contract-required detection limit 10. A control limit of \pm the contract required detection limit shall be used if either the sample (S) or the duplicate value (D) is less than 5 times the contract required detection limit: $RPD = \frac{S-D}{[(S+D)/2]} \times 100$ 11. Verify from the raw data that the appropriate number of required duplicate samples were prepared and analyzed and that duplicate results, for each analyte and method, fall within the established control limits <p>Different types of duplicate samples are</p> <ol style="list-style-type: none"> 12. Collocated samples (sampling and measurement precision) 13. Field replicates (precision of all steps after collection) 14. Field splits (shipping and inter-laboratory precision) 15. Laboratory splits (inter-laboratory precision) 16. Laboratory replicates (analytical precision) 17. Analysis replicates (instrument precision) <p>Results of field duplicate sample analyses have generally higher variability than the results of laboratory duplicate analysis because laboratory duplicates measure laboratory performance, while field duplicates reflect the difficulties associated with collecting identical field samples</p>	Laboratory Technical Personnel
Spike Sample Analysis	<p>Verifies that the following spiked samples were performed to provide information on the effect of each sample matrix on the sample preparation procedures and measurement methodology:</p> <ol style="list-style-type: none"> 18. Matrix spike (spiked prior to digestion) 19. Analytical spike (spiked after the completion of the distillation or digestion procedure) <p>Also verifies the following:</p> <ol style="list-style-type: none"> 20. Percent recoveries associated with spiked samples were within established acceptance/control limits (does not apply when sample concentration exceeds the spike concentration by a factor of 4 or greater) 21. The appropriate number of required spike samples was prepared and analyzed 22. Spiked sample results were within established control limits 23. The field blank was not used for the spiked sample analysis <p>Spiked sample analysis also measures system bias as indicated below:</p> <ol style="list-style-type: none"> 24. Matrix spike: analytical (sample preparation and analysis) bias 25. Matrix spike replicate: analytical bias and precision 26. Analytical matrix spike: instrumental bias 27. Surrogate spike: analytical bias 	

Table 3-1. Examples of Data Compliance Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
Laboratory Control Sample (LCS) Analysis	28. Reviews preparation logs, raw data, instrument printouts 29. Serves as a monitor of the overall performance of each step during the analysis, including sample preparation 30. If the percent recovery for LCS falls outside of fixed control limits, the analysis should be terminated, the problem corrected, and the samples associated with that LCS re-digested and analyzed 31. Verifies that appropriate number of LCS were analyzed and results were within acceptable control limits	Laboratory Technical Personnel
Confirming Calibration Verification Calculations	Uses the following methods to verify a calibration curve fitting: <ul style="list-style-type: none"> Verify that the laboratory-reported percent recovery for the initial calibration verification (ICV) standard or continuing calibration verification (CCV) standard equals the ratio of the reported value to the true value (expressed as a percentage) and is within acceptable limits Verify that the ICV and CCV standards were analyzed for each analyte at the proper frequency and at the appropriate concentration 	QA Administrator or QA Auditor
Method Detection Limits Audit	Confirms that the analytical system to be used can meet the specified method detection limit as given in the MQOs	
Calibration Standard Preparation Audit	Verifies the date and time that analytical standards were prepared	
Calibration Corrective Action Audit	Verifies that the appropriate corrective action was taken if calibration or calibration check data failed to meet acceptance criteria	
Sample Preservation and Handling	Verifies the integrity of the sample and ensures that the sample underwent proper receipt and handling procedures; documents that the proper preservation of the sample was maintained (e.g., temperature and preservatives)	Sample Custodian
Sample Storage	Ensures that holding times for sample storage were met	

Table 3-2. Examples of Data Correctness Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
Instrument Inspection and Maintenance	Ensures that all analytical instruments, sampling equipment, etc., were maintained in proper operating condition by reviewing instrument maintenance and inspection logs	Field and Laboratory Technical Personnel
Instrument Calibration Audit	Establishes the relationship between the actual pollutant concentration and the instrument's response: <ul style="list-style-type: none"> Establishes method requirements for satisfactory instrument calibration to ensure that the instrument was capable of producing acceptable quantitative data Ensures calibration was performed within an acceptable time prior to generation of measurement data Verifies that calibration was performed in proper sequence Ensures that calibration was performed using standards that bracket the range of reported measurement results 	Laboratory Technical Personnel

Table 3-2. Examples of Data Correctness Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
	<ul style="list-style-type: none"> Ensures acceptable linearity checks to ensure the measurement system was stable when calibration was performed Checks preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data Confirms the linearity of the calibration curve was within acceptable limits (e.g., $R > 0.995$) Confirms that one of the calibration standards was analyzed at the required detection limit 	
Data Recording Audit	Verifies the internal checks used to ensure data quality during data encoding in the data entry process and the mechanisms for documenting and correcting recording errors	QA Administrator or QA Auditor
Data Reduction Audit	Reviews the audit trail of the data reduction process	
Data Transformation Audit	Verifies all data transformations and reviews conversion of calibration reading into equations applied to measurement readings	
Raw Data Audit	<ul style="list-style-type: none"> Examines raw data to verify that the correct calculation of the sample results were reported by the laboratory Examines raw data for any anomalies Verifies that there are no transcription or reduction errors 	

Table 3-3. Examples of Data Consistency Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
Data Handling Audit	Ensures the accuracy of data transcription and calculations by checking a set of computer calculations manually	QA Administrator or QA Auditor
Data Transmittal Review	Reviews the data transfer steps and the procedures used to characterize data transmittal error rates in order to minimize loss in the transmittal	

Table 3-4. Examples of Data Completeness Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
Chain of Custody (COC) Documentation	Documents the progression of samples as they travel from the original sampling location to the laboratory and finally to their disposal; ensures proper use of COC seals	Sample Custodian, Field and Laboratory Technical Personnel
Sample Records Documentation and Audit	Ensures that an accurate written record was maintained of sample handling and treatment from the time of collection through laboratory procedures to final disposal	Field and Laboratory Technical Personnel

Table 3-4. Examples of Data Completeness Procedures and Their Purpose

Procedure or Verification Step	Purpose	Person(s) Responsible
Instrument Inspection and Maintenance Documentation	Documents the capability of the instruments to perform the necessary operations	
Sample Shipment Documentation	Ensures proper documentation of Chain of Custody and sample handling during transfer from the field to the laboratory, within the laboratory, and among contractors	Sample Custodian and QA Administrator
Data Management Audit	Traces the path of the data from their beginning in the field or laboratory to their final use or storage	QA Administrator or QA Auditor
Documentation of QC Results including Control Charts and Audit	Documents general QC measures such as initial demonstration of capability, instrument calibration, routine monitoring of analytical performance, calibration verification, etc., and reviews established QC warning and control limits for the statistical data generated by QC checks	Field and Laboratory Technical Personnel, QA Administrator
Sample Log In	Verifies COC, documents problems such as receipt of damaged samples, and records proper log-in of samples into the laboratory	Sample Custodian
Sample Identification Audit	Ensures that a unique identification number was assigned to each sample	QA Administrator
Documentation of Field Corrective Action	Shows what methods were used in cases where general field practices or other standard procedures were violated	Laboratory Technical Personnel
Traceability of Standards Review	Documents standards' traceability relative to a certified, reproducible reference point	
Documentation of Calibration Corrective Action	Documents actions taken if a QC check identifies a failed or changed measurement system	
Laboratory Analysis Records Review	Ensures that the appropriate analytical method was used and that any failure in the analytical system was properly documented, and evaluates the effectiveness of the corrective action	
Documentation of Laboratory Corrective Action	Shows what methods were used in cases where general laboratory practices or other standard procedures were violated	

through 3-4. Thus, most laboratories are well informed on implementing the data verification procedures. As a result, details on how each of the data verification procedures should be implemented in an environmental data collection program are not provided. Instead, examples of how these procedures may be implemented within the case study presented in Section 2.3 are given.

The following subsections discusses each of the four data verification elements in the context of Tables 3-1 through 3-4, with examples from the case study introduced in Section 2.3.

3.1.1 Compliance

Note that the compliance procedures included in Table 3-1 address such issues as the following:

- staff responsibilities and qualifications,
- field and laboratory audits of sample collection/preparation and corrective actions,
- QC sample collection and analysis (field and laboratory),
- efforts to verify the calibration curve used to quantify analyte concentrations, and
- sample handling and storage.

*Case Study Example of Selecting and Applying
Compliance Procedures*

In the case study on emissions sampling introduced in Section 2.3, the project manager has reviewed Table 3-1 and indicates that the following compliance procedures will be among those incorporated into the project's data verification process. Each procedure is accompanied by its planned approach.

- Staff Training and Certification: The Project Manager will provide initial training in Method 29 sample collection procedures to all field personnel and document it in each individual's training record that is kept in centralized department files. Certificates will be issued to individuals with specialized training and copies retained for their file. Written procedures will be provided to field staff and posted in sampling areas for reagent and train preparation.
- Sample Custodian Assignment: The Project Manager will assign a Chain-of-Custody (COC) Officer or Sample Custodian to track samples sent back or brought from the field. This person will have complete control over access to samples and distribute samples to the appropriate analytical staff after confirming COC forms were filled in properly and samples were in good condition upon receipt at the laboratory.
- Field Data Collection Audit: The QA Auditor will perform an internal audit of sample collection procedures and documented findings by issuing a corrective action report. The audit will consist of observing sampling and sample handling procedures to evaluate adherence to the QAPP and field SOPs. The QA Auditor will also review the completed Method 29 field sampling data sheets to ensure that all information has been recorded and that any technical problems are discovered and addressed. An audit report to the Project Manager will detail any discrepancies found.

- Method Blank Analysis: During sampling events, field technical personnel will collect samples of all rinse solutions at least once for each sampling train for use as method blanks. These QC samples will be used to verify that samples are not contaminated at any point throughout the sample collection process. These will be collected at the appropriate interval specified in the QAPP.
- Blank Sample Collection and Analysis: Field technical personnel will be responsible for the collection of blank samples at the interval specified in the QAPP. Field blanks will be collected for each sampling train and sample type and transported to the laboratory with the samples. An aliquot of each reagent will also be collected to verify that reagents are not contaminated. During sampling events, field technical personnel will collect samples of all rinse solutions at least once for each sampling train for use as method blanks. These QC samples will verify that the sampling equipment does not contaminate samples. These will be collected at the appropriate interval specified in the QAPP.
- Replicate Sample Collection and Analysis: The Field Team Leader will be responsible for ensuring on a daily basis that sampling is conducted using EPA Method 29 and that triplicate emission data are collected for assessment of variability due to sampling of metals. This will be accomplished by the collection of triplicate field samples.
- Calibration: Calibration of field instruments will be performed by the field technical personnel prior to sample collection and documented in the calibration logbook. Calibration will also be performed immediately following sample collection and the results of the calibration will be required to be within 5% of the initial calibration or the data collected with that piece of equipment was to be flagged. The Field Team Leader will be responsible for verifying that the initial and final calibrations are within the limits specified in the QAPP. The calculations will be reviewed and any mistakes found will be corrected and documented.
- Confirming Calibration Verification Calculations: The Field Team Leader will be responsible for review of all verification checks performed during calibration of field equipment. Staff performing equipment calibrations will use a spreadsheet template that prompts the field technician for the appropriate information. Linearity checks and calibration verifications will be calculated automatically by formulas within the spreadsheet.
- Calibration Corrective Action Audit: The Field Team Leader and QA Administrator will verify that when problems occur during calibration of field equipment, appropriate corrective actions are taken and documented. Field data sheets and instrument calibration logbooks will be reviewed as part of an internal technical systems audit.

- Sample Preservation and Handling: Field technical personnel will be responsible for following proper sample preservation and handling requirements during collection as described in the QAPP and field SOPs. The Sample Custodian will be responsible for maintaining these requirements through preparation for and shipment to the laboratory. For example, sorbent traps will be sealed immediately by field personnel upon disassembly of the sampling unit and stored at 4 °C until they are relinquished to the Sample Custodian. The Sample Custodian will document that all the samples were in compliance upon receipt and prepare the samples for shipment to the laboratory according to the SOP by sealing them in charcoal-containing canisters.
- Sample Storage: The QA Administrator will inspect field sampling data sheets and chain of custody documentation and survey actual sample storage procedures for comparison with protocol in the QAPP and field SOPs to verify proper storage of samples in the field. The coolers will be inspected, and the QA Administrator will verify that cooler thermometers are traceable to a NIST standard thermometer. Thermometer calibration documentation and logbooks will be reviewed to ensure adherence to SOPs. Discrepancies will be discussed with the Field Team Leader during the audit to identify proper corrective action. Sample storage audit findings will be reported with the technical systems audit report to the Project Manager.

3.1.2 Correctness

The correctness procedures included in Table 3-2 address such issues as the following:

- audits of instrument calibration and data audits, and
 - instrument inspection and maintenance.
-

*Case Study Example of Selecting and Applying
Correctness Procedures*

In the case study from Section 2.3, the project manager plans to implement the following correctness procedures within the project's data verification process:

- Instrument Inspection and Maintenance Audit: The Field Team Leader will inspect instrument maintenance logs before initial calibration of field equipment to verify that proper maintenance procedures have been followed, document any findings, and issue a corrective action report to the QA Administrator.
- Instrument Calibration Review: The Field Team Leader will verify that dry gas meters are calibrated just prior to the departure of the equipment to the field, a post test calibration is performed, and both calibrations agree to within 5 percent.

Field technical personnel will verify that analytical balances are calibrated over the expected range of use with NIST Class S standard weights and that this is properly documented in a calibration logbook. The Field Team Leader will verify that field technical personnel report calibration problems when they occur, and the results that are questionable, in order to quality the significance of that data.

- Data Recording Audit: The QA Auditor will review data recording procedures during the technical systems audit. Throughout the project, field technical personnel will complete a sample data sheet for each impinger that contained original and final volumes of reagent in each impinger. Field technicians will measure the volume of impinger contents by weighing to nearest gram before and after sampling and calculate the difference. The QA Auditor will verify internal checks to ensure these data are recorded properly onto sample data sheets and will review the mechanisms for documenting and correcting recording errors by observing data recording and comparing to SOPs.
- Data Reduction Audit: The QA Auditor will be responsible for reviewing the audit trail of the data reduction process. A Method 29 data reduction spreadsheet will be used to generate final gas sample volumes at standard conditions, which is used to calculate trace element emission rates. The QA Auditor will repeat the calculation manually for three samples to verify that the appropriate data reduction procedure was used.
- Data Transformation Audit: The QA Auditor will review the process of data transformation during the technical systems audit. Field technicians will perform and document manual data reduction using a calculator and an electronic data reduction program. The data reduction program will compute gas sample volume, moisture content, stack gas molecular weight, velocity, flow rate, and isokinetic ratio.
- Raw Data Audit: The Field Team Leader will be responsible for review of raw data on all sample collection and on non-routine process parameters, such as hopper loads and catch rates, to determine the consistency of the data for the period of the test. Outliers will be flagged. Field technical personnel will review raw data on sample collection data sheets for completeness and transcription errors.

3.1.3 Consistency

The consistency procedures included in Table 3-3 address such issues as the following:

- data handling audit, and
- review of data transmittal efforts.

*Case Study Example of Selecting and Applying
Consistency Procedures*

In the case study introduced in Section 2.3, the project manager has reviewed Table 3-3 and has included the following consistency procedures in the project's data verification process:

- Data Handling Audit: The Field Team Leader will be responsible for verifying proper data handling procedures were followed. During the project, site staff will provide plant operation data in the form of copies of standard computer printouts from an automated data system. The Field Team Leader will review the data with the plant manager to ensure that the reports are accurate and represented the appropriate time period. Field staff will generate hand-written data sheets for each sample collected during the project that contain pump flow rates and on/off times. Each field technician will be responsible for checking 100% of hand-written data for transcription errors.
- Data Transmittal Review: All data will be transferred to the laboratory on hand-written data sheets, along with the samples.

3.1.4 Completeness

The completeness procedures included in Table 3-4 address such issues as the following:

- proper chain-of-custody
- maintenance and review of sample records
- documentation of all aspects of sample handling, data reporting, and corrective action taken

*Case Study Example of Selecting and Applying
Completeness Procedures*

In the case study introduced in Section 2.3, the project manager has reviewed Table 3-4 and indicates that the following completeness procedures will be among those incorporated into the project's data verification process:

- Documentation of Field Corrective Action: The Field Team Leader and all field staff will be responsible for documenting any problems that occur during sample collection in the field. If corrective action must be taken, it will be the responsibility of the field staff to document these actions and their potential impact on the data.

- Sample Records Documentation and Audit: The field technical personnel will be responsible for completing Method 29 field sampling data sheets to document sampling times, temperature, person performing the sampling, sampling location, and sampling points. Field personnel will also utilize the established sample identification system and label all samples at time of collection. Field data sampling sheets will serve as the COC form and will become the responsibility of the Sample Custodian as soon as the samples are collected and labeled. All information relevant to verify sample integrity will be recorded on the sheets including sample preservation, storage, and shipment information.
- Sample Shipment Documentation and Audit: The Field Team Leader will be responsible for verifying that Chain of Custody forms remain with the samples during shipment to the laboratory and that the appropriate method of shipment is used and documented. The Sample Custodian and QA Auditor will review documentation to ensure appropriate handling and preservation requirements are met.
- Data Management Audit: The QA Auditor will be responsible for review of data management procedures used in the field during this project as part of an internal technical systems audit. This will be done by tracing sample documentation from the time it is collected through the time it is shipped to the laboratory for analysis. The records reviewed will include sample collection data sheets and COC forms.
- Chain of Custody Documentation: Field technical personnel will complete a COC form accompanying every flue gas sampling train, specific for a given sample and sampling location from the moment the train is assembled. It will be signed by the person performing the sampling and relinquished to the Sample Custodian along with the samples for shipment to the laboratory. The Sample Custodian will be responsible for ensuring that all applicable procedures are followed for preservation, handling, and shipment to the laboratory and that required information and deviations were documented on the COC form. The COC form will remain with the samples during shipment to the laboratory.
- Sample Identification Audit: The QA Auditor will conduct a sample identification audit as part of the internal Technical Systems Audit that is required by the QAPP for the project. The QA Auditor will review labeled sample containers and COC forms with the Sample Custodian to ensure that each sample has been assigned a unique sample identification and that this identification is properly documented on the COC.
- Instrument Inspection and Maintenance Documentation: Field technical personnel will be responsible for ensuring that all sample collection equipment used in the field is checked daily for operational capability and alignment prior to field

operations and is documented according to EPA Method 29 criteria. A list of available spare parts will be maintained according to inspection and maintenance SOPs and these activities will be documented in the field equipment inspection and maintenance logbook. Other maintenance that is required on a semi-annual basis will be assigned to field personnel and documented in the same logbook.

- Traceability of Standards Review: The Field Team Leader will be responsible for the review of standard certificates and preparation logs to ensure that standards meet requirements for traceability to national standard reference materials (SRMs).
 - Documentation of Calibration Corrective Action: The field technical personnel will be responsible for ensuring that any problems and corrective actions taken during field instrument calibration are documented in the field equipment calibration logbook. All equipment will be calibrated for the field operation before traveling to the sample collection site.
-

3.2 VERIFYING THAT MQOs HAVE BEEN MET

Figure 1-3 of Chapter 1 implies that an important objective of the data verification process is to determine the extent to which pre-specified measurement quality objectives (MQOs) have been met. MQOs are specific goals that clearly describe the performance requirements for a measurement system. MQOs specify acceptance criteria for Data Quality Indicators, such as selectivity, sensitivity, detection limits, bias, precision, representativeness, comparability, and completeness for the collected data.

- Detection limit (DL): Specified within the MQOs prior to sample analysis. Types of detection limits include instrument detection limits (lowest concentration of an analyte that an instrument could reliably distinguish between signal and background), target detection limits (lowest concentration that can be used to reliably assess and satisfy DQOs), method detection limits (ability of the method to detect the analyte in the sample matrix regardless of its source of origin), and limit of quantification.
- Bias: Expected difference between the “true” value of the analytical concentration within a sample and what the analytical method reports as the measured concentration of the analyte in the sample.
- Precision: Expected level of agreement among multiple measurements of the same characteristic.
- Representativeness: The degree to which the collected data accurately represent the environment being monitored by the project. Representativeness is maintained

by following procedures such as complying with a statistically-based field sampling design, proper sample storage, and proper sample homogenization.

- Comparability: Expected level of confidence with which data sets from different

Data Verification Checklists

During the verification procedure, checklists are used frequently by the QA Auditor and other project staff to ensure that all critical elements of sample and data collection and reporting have been addressed and resolved. The checklists should identify critical verification elements and should document the result of verification activities. Checklists are based on the requirements of the QAPP and should therefore include verification elements for assessing compliance with:

- analytical methods,
- standard operating procedures,
- DQOs, and
- general quality systems associated with each.

The best checklists combine project-specific requirements that are defined in the QAPP with method or SOP references. Checklists should “walk” the verifier through the data collection process. Tables 2-1 to 2-4 provide examples of the types of information that should be verified. Table 3-5 illustrates example checklist entries for compliance procedures documented within Table 3-1 above and performed within the case study presented in Section 2.3 and the two additional case studies presented within Appendix C.

**Table 3-5. Example Checklist Entries for Compliance Procedures,
As May Occur Within the Three Case Studies**

Verification Item	Citation	Example Checklist Entry
Field instrument inspection and maintenance (Case study presented in Section 2.3)	EPA Method 29	The field equipment inspection and maintenance logbook documented that the operation and alignment of sample collection equipment was checked daily and documented according to EPA Method 29 criteria.
	SOP 1	A list of available spare parts was maintained according to inspection and maintenance SOPs and these activities were documented in the field equipment inspection and maintenance logbook.
Laboratory control sample (Case Study C.1, Appendix C)	QAPP	The percent recovery limits established in the program QAPP for LCS=100 ± 10%. During an analysis, the LCS=78% for Cadmium and 76% for Copper. The analysis was terminated and the samples associated with that LCS were re-digested and analyzed.
Control Charts (Case Study C.2, Appendix C)	EPA 600/4-90/027F	The reference test was run monthly and all records maintained in a reference test log book. Each successive LC50 was documented on the control chart. It was not always possible to trace the preparation of stock solution used in the reference test because the preparation was not documented each month. The laboratory technical personnel documented that the same dilution scheme was used each month.

Data Verification Reports

As illustrated in Tables 3-1 through 3-4, several members of a project team are responsible for data verification activities. Some of the data verification procedures are the responsibility of

the technical personnel, while other verification activities are the responsibility of the QA Administrator's staff.

At the technical levels (field technical personnel, Field Team Leader, data analyst, laboratory manager), verification involves reviewing daily activities and comparing the findings to the requirements of the QAPP, methods, and SOPs. The results of technical verification typically result in:

- confirming that the outcome is acceptable and that no corrective action is needed;
- identifying missing or additional documentation that can be retrieved and included;
- identifying minor deviations that are corrected if possible;
- identifying major deviations that are immediately reported to the Project Manager; and
- documenting that corrective action was taken.

Technical verification may be documented on internal checklists that are signed and dated; formal reports may not be required.

Verification activities performed by the QA Administrator are independent of verification at the technical level. It is the responsibility of the QA Administrator to relay the results of data verification procedures to the project manager. This can take the form of a brief report that presents and discusses the results of the quality control data and identifies deviations from the QAPP, analytical method, or SOP requirements. The report should also identify problems that should be investigated during the data validation process (Chapter 4) because they could affect the usability of the data. Results can also be presented informally, upon agreement with the project manager.

The outline of the data verification report should include the following:

<p>1. PROJECT INFORMATION</p> <ul style="list-style-type: none">• Project title• Client• Project Manager• Date of Study Verification• Personnel Responsible for Phase Being Verified
<p>2. DATA VERIFICATION (For each element included in this verification the following issues should be addressed: compliance, documentation).</p> <ul style="list-style-type: none">• Sample collection• Field measurements• Sample custody, preservation, holding times, and tracking• Laboratory processing• Instrument calibration, maintenance, and analysis• Data reduction and reporting• Quality control results

3. CORRECTIVE ACTION (For each finding in Section 2 the responsible party must document the corrective action).
4. APPROVALS QA Administrator _____ Date _____ Project Manager _____ Date _____

Verified Raw Data Package

When required, the raw data package should present all data and supporting documentation needed to reproduce the results of data collection. The specific contents of the raw data package will depend upon the types of samples collected and analyzed. Two examples are presented below to illustrate typical contents of a raw data package.

Raw data packages for studies that collect organic or inorganic data (e.g. the case study in Section 2.3 above and Case Study C.1 in Appendix C) may include:

- client and laboratory identification numbers;
- sample collection date;
- sample matrix;
- date of sample extraction;
- date and time of analysis;
- identification of instrument used for analysis;
- instrumental specifications (e.g., GC column and detector);
- sample weight or volume;
- dilution or concentration factor;
- method detection limits or quantification limits;
- analytical results and associated units;
- definitions for any laboratory data qualifiers used;
- initial calibration summaries;
- continuing calibration summaries;
- method blank results;
- surrogate percent recoveries;
- matrix spike percent recoveries;
- laboratory duplicate relative percent differences;
- laboratory QC check sample;
- retention times and acceptance windows;
- results for the environmental samples (including re-analyses);
- instrument tuning;
- initial calibration results;
- continuing calibration results;
- method blank results;
- surrogate recovery results;
- laboratory duplicate or matrix spike duplicate results;

- laboratory QC check sample results;
- sample extraction and clean-up logs;
- instrument analysis logs; and
- sample custody records.

For toxicity tests (e.g. Case Study C.2 in Appendix C) the raw data package may include:

- taxonomic identification of organism;
- client and laboratory identification numbers;
- sample collection date and time (initial and final), and collection method;
- sample holding temperature;
- test type and duration;
- test temperature;
- light quality/intensity and photoperiod;
- test chamber size, test solution volume;
- schedule of test solution renewal;
- species tested and age of species;
- number of organisms per test chamber, replicate chambers per concentration, number of organisms per concentration;
- test conditions and maintenance: test chamber cleaning, test solution aeration, feeding regime;
- description of dilution water;
- test concentrations and dilution series;
- control percent survival;
- reference test results with upper and lower control limits;
- water quality average and range during test (pH, DO, salinity, temperature);
- sample custody records;
- test species data: identification, culture records, hatch and acclimation records;
- food lot analysis results;
- dated dilution water analysis preparation records and analysis results;
- reference test data: solution preparation, bench sheets of daily survival and water quality measurements, control charts;
- sample characterization, adjustments, and dilution scheme;
- test initiation data (initial water quality, mysid observations and randomization);
- instrument calibration records;
- daily animal observations, water quality, cleaning, aeration, and feeding records;
- final tally of survival in each control; and
- probit analysis and LC50 determination.

3.4 AUTOMATED DATA VERIFICATION

While data verification has historically been performed manually, more recent efforts have allowed data verification to become more automated. Commercially-released computer software exists that can perform some of the data verification procedures discussed in this chapter. For example, many data verification software products exist that automatically perform data correction procedures, primarily on data that were hand entered. Information about the use of automated data verification software can be found in EPA's Good Automated Laboratory Practices (1995).

Prior to the use of any automated data verification software, it is imperative that the software has been completely validated to ensure that it accomplishes the procedures completely and correctly and that it is well documented. For example, software verification is vital for regulatory agencies involved in risk assessment using computerized software programs. Software validation will have been done for most of the commercially available software programs, but it may not have been done for data verification software that is not available commercially. EPA has not formally evaluated automated data verification software and does not endorse a specific brand of software. Ultimately, responsibility for validation of the software lies with the user. Information on software validation is provided in Appendix D.

(This page left blank intentionally.)

CHAPTER 4

IMPLEMENTING THE DATA VALIDATION PROCESS

The data validation process consists of the following:

- Ensures that the measurement system (field and laboratory) meets the users' needs.
- Assigns qualifiers to individual data values based on whether the analyte in question is detected and the associated degree of variability, with consideration given to the level of deviation from performance standards.
- Assesses the relevancy of certain performance criteria used to make decisions on the observed data, given information obtained during the course of the project.
- Determines whether the data can proceed to Data Quality Assessment (and the evaluation of whether DQOs were satisfied).

As seen in Figure 1-2 of Chapter 1, data validation is begun once the data have successfully passed the data verification process discussed in Chapter 3. While data verification addresses the extent to which specific procedures specified in the QAPP were followed and certain criteria were met, data validation focuses on the ability to use the data as intended to make decisions and to address project objectives (e.g., for the case study presented in Section 2.3, to evaluate emission control devices at a coal-fired power plant). Therefore, data validation centers on evaluating the extent to which the collected data provide the necessary information to meet the needs of the project's stakeholders. Data validation is typically performed by persons not directly involved with generating the data. This chapter discusses various components of the data validation process.

In data validation, each reported data value is assigned a qualification indicating the degree to which the reporting of this value deviated from performance criteria. Equivalently, the qualification of a data value measures the degree to which the value can be utilized as intended when making conclusions based on the entire set of data. These qualifications address overall usability, not contractual adherence. Examples of data qualifications include:

- analyte is not detected above the method detection limit,
- quantity of analyte is approximate due to analysis limitations,
- identification of the analyte is tentative,
- identification of the analyte is uncertain (with reason given, such as interference), and
- quantity of analyte is confirmed.

1 The datum qualification may be affected by several types of outcomes, including holding time,
2 sample condition, and QA and QC analysis results. Each outcome that may affect the usability of
3 a data value must be considered by the data validation procedure. Note that assigning data
4 qualifications may involve using information obtained by executing the data verification
5 procedures discussed in Chapter 3. For example, a finding that sample collection, handling, or
6 analysis procedures were not properly followed may result in flagging the affected data value as
7 not to be used or as containing considerable uncertainty. Data validation, however, should not be
8 restricted to only those data identified as suspect in the data verification procedures. Each data
9 value should be examined to determine the extent to which it should be qualified.

10 When an analyte is detected within a sample and the concentration is reported, data
11 validation also involves assigning a data qualifier to the result according to the level of uncertainty
12 associated with the reported value. This procedure typically evaluates whether the data value falls
13 within specified confidence limits that are determined from pre-specified variability assumptions.

14 QA and QC considerations also can enter into the data qualification procedure. Examples
15 of such considerations are given in the context of the case study on emissions monitoring
16 (Section 2.3):

- 17 • The data validation process would consider whether EPA Method 29 was followed
18 to collect the air samples. If deviations from this method occurred, such as use of
19 the wrong sampling equipment or improper preservation of air samples during

1 Data validation also involves reviewing whether the performance criteria (e.g., MQOs)
2 were specified adequately or appropriately within the QAPP. This review is done utilizing
3 information that was not available when performance criteria were specified in the QAPP, but was
4 identified during the course of the project. For example, information on the magnitude of
5 analytical error reported in the project may result in re-evaluating certain criteria placed on
6 precision.

7 In general, evaluating the extent to which the project DQOs have been satisfied by the
8 data is done during the Data Quality Assessment, which follows data validation. Therefore,
9 evaluating the DQOs is generally not a part of the data validation process. However, planning the
10 data validation process should be done in parallel with the DQO Process to ensure that validation
11 can be done efficiently with regard to the eventual Data Quality Assessment. In some instances,
12 information existing within the DQOs can actually be used in certain aspects of data validation.
13 For example, in the case study on emissions monitoring (Section 2.3), the DQOs indicate that a
14 minimum quantity of emissions must be sampled in order to obtain sufficient information for
15 comparison purposes and for calculating removal efficiencies. This relates to a sample's volume,
16 which may need to achieve a certain magnitude before analytes can be successfully detected
17 within the sample according to MQOs for the method detection limit. If too little air is sampled,
18 the method detection limit may be higher than the level of quantification, and the data would not
19 be usable for the study. Therefore, the information within these DQOs may contribute to
20 establishing the criteria for a minimum sample volume that would be used in assigning data
21 qualifications within the data validation process.

22 Typically, data validation must be performed by knowledgeable individuals who can best
23 perform evaluations within the various validation components listed above. Automation is
24 generally not applicable to data validation, although it is appropriate for data verification (see
25 Section 3.4).

26 In some cases, contractual requirements may specify that data validation be performed by
27 an independent, third party validator. Besides the project objectives, the validator would need the
28 information obtained from the data verification procedures in order to assign additional data
29 qualifiers based on applying its validation procedures.

30 Once the data validation process has been completed, the verified and validated data are
31 ready to be used as input for the Data Quality Assessment stage of the EPA Quality System.

4.1 REPORTING THE RESULTS OF DATA VALIDATION

It is the responsibility of the QA Administrator to relay the results of data validation procedures to the Project Manager and to characterize the level of confidence that is being placed in the data and the data collection process. While this would take the form of a brief report, results can also be presented informally, upon agreement with the Project Manager. The validation report should include:

- a cover page;
- an assessment of the overall test results and a determination of the usability of the data;
- summary of environmental sample results; and
- summary of QA and QC results.

The specific elements and format required for the data validation report (whether informal or detailed) should be determined prior to the start of the project and specified in the QAPP. Possible contents of these items are discussed below.

Cover Page

The report should have a formal cover page that identifies the project title, client, organization responsible for the generation of the data, report date, and the person(s) and organization or agency responsible for completing the validation.

Assessment of Data Usability

The assessment of the usability of the test results is the final product of the data validation process. The QAPP must define the type of assessment required, who is responsible for performing the assessment, and how the results of the assessment will be reported. Options include one or more of the following:

- Assignment of data qualifiers by the laboratory,
- Validation of laboratory data by an independent (third party) validator; assignment of validation qualifiers in addition to those assigned by the laboratory, and
- Validation by the regulatory agency; preparation of a validation report that identifies the data as acceptable, acceptable with condition (or correction), or unacceptable.

Although the criteria for acceptance of the validated results are specified in the QAPP, the experience of the validator weighs heavily in these decisions when the criteria are not well-defined or where judgements are necessary.

The assessment of data usability should also discuss any discrepancies between the DQOs and the data collected and any effects the discrepancies may have on the attainment of the DQOs.

Summary of Environmental Sample Results

A summary of environmental sample and data collection results is the third item in the data validation report. This information is typically provided as report tables or spreadsheets. Information in this report may include:

- client and laboratory identification numbers;
- sample matrix;
- sample collection date;
- sample extraction date;
- sample analysis date;
- sample extraction and/or analysis methods;
- identification of instrument used for analysis;
- instrument specifications;
- sample weight or volume;
- dilution or concentration factor;
- analytical results and associated units;
- qualifiers that are applied during verification and validation;
- method detection limits or sample quantification limits; and
- definitions for any laboratory data qualifiers used.

Summary of QA and QC Results

The last item in the data validation report is a summary of the QA and QC results. The results of all quality control samples required by the QAPP must be reported. Example results to be included in the QA and QC summary are as follows:

- sampling and analytical precision (field and/or laboratory replicates);
- analytical accuracy [surrogates, laboratory control samples (LCSs)], matrix spike samples, SRMs;
- decontamination and cross-contamination assessment (field, shipping, method blanks);
- test animal health (toxicity test control results);
- test animal sensitivity (toxicity test reference tests);
- method conformance (summary of analytical procedures, toxicity test physical testing conditions);
- a narrative that discusses any deviations from the QAPP, including quality control failures, and the impact of those failures on the data.

1 If the data validation report is part of a complete data validation package then a full,
2 verified copy of the raw data is included. The format and contents of the raw data package are
3 described in Section 3.3.

4 REFERENCES

5 Karnofsky, J. (1997). *Radiochemistry Data Validation, Version #7*.

6 U.S. Environmental Protection Agency (1993) *Methods for Measuring the Acute Toxicity of*
7 *Effluents to Freshwater and Marine Organisms, Fourth Edition*. Environmental
8 Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati,
9 Ohio. August 1993. EPA/600/4-90/027F.

10 U.S. Environmental Protection Agency (1994) *Assessment and Remediation of Contaminated*
11 *Sediments (ARCS) Program: Assessment Guidance Document*. Final Draft. Oceans and
12 Coastal Protection Division, U.S. Environmental Protection Agency, and Great Lakes
13 National Program Office. July 1994.

14 U.S. Environmental Protection Agency (1995) *Good Automated Laboratory Practices:*
15 *Principles and Guidance to Regulations for Ensuring Data Integrity in Automated*
16 *Laboratory Operations*. Office of Information Resources Management, U.S.
17 Environmental Protection Agency, Research Triangle Park, North Carolina. August,
18 1995. EPA 2185.

19 U.S. Environmental Protection Agency (1998) *EPA Guidance for Quality Assurance Project*
20 *Plans, EPA QA/G-5, EPA/600/R-98/016*. Washington, DC.

21 U.S. Environmental Protection Agency (1998) *Guidance for Data Quality Assessment: Practical*
22 *Methods for Data Analysis, EPA QA/G-9, EPA/600/R-96/084*. Washington, DC.

23
24 U.S. Environmental Protection Agency (1999) *EPA Requirements for Quality Management*
25 *Plans, EPA QA/R-2, EPA/600/XXX/XXX*. Washington, DC.

26 U.S. Environmental Protection Agency (1999) *EPA Requirements for Quality Assurance Project*
27 *Plans, EPA QA/R-5, EPA/600/XXX/XXX*. Washington, DC.

APPENDIX A

GLOSSARY OF TERMS

These definitions were obtained from various sources referenced at the end of the definitions. The number of the reference(s) is given in parentheses at the end of the definition. In instances where several sources contained multiple definitions of terms, the definition given reflects a consensus definition.

acceptance criteria — specified limits placed on characteristics of an item, process, or service defined in requirements documents. (ASQC Definitions) (12)

acceptance windows — the quantitative range that is between the lower acceptance limit and the upper acceptance limit. (11)

accuracy — a measure of the closeness of an individual measurement or the average of a number of measurements to the true value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; the EPA recommends using the terms “precision” and “bias”, rather than “accuracy,” to convey the information usually associated with accuracy. (12)

activity — an all-inclusive term describing a specific set of operations of related tasks to be performed, either serially or in parallel (e.g., research and development, field sampling, analytical operations, equipment fabrication), that, in total, results in a product or service. (7, 12)

analyte — a target analyte is an environmental compound or element that is being measured or identified in a chemical test to satisfy project-specific data objectives. Target analytes are distinguished from compounds or elements analyzed solely for the purposes of quality control (e.g., surrogates, matrix spikes and laboratory control samples). For brevity, target analytes are often referred to as analytes.

assessment — the evaluation process used to measure the performance or effectiveness of a system and its elements. As used here, assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, management systems review, peer review, inspection, or surveillance. (5,7,12)

audit (quality) — a systematic examination to determine whether quality activities, systems and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives. (9)

auditor — a person qualified to perform audits. (9)

bench sheets — the forms used to record routine measurements and observations for toxicity tests. (11)

bias — the systematic or persistent distortion of a measurement process which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (7)

blank — a sample subjected to the usual analytical or measurement process to establish a zero baseline or background value. Sometimes used to adjust or correct routine analytical results. A sample that is intended to contain none of the analytes of interest. A blank is used to detect contamination during sample handling, preparation, and/or analysis. (12)

calibration drift — the deviation in instrument response from a reference value over a period of time before recalibration. (12)

calibration — comparison of a measurement standard, instrument, or item with a standard or instrument of higher accuracy to detect and quantify inaccuracies and to report or eliminate those inaccuracies by adjustments. (4)

certification — the process of testing and evaluation against specifications designed to document, verify, and recognize the competence of a person, organization, or other entity to perform a function or service, usually for a specified time. (12)

chain of custody — an unbroken trail of accountability that ensures the physical security of samples, data, and records. (7)

characteristic — any property or attribute of a datum, item, process, or service that is distinct, describable, and/or measurable. (1)

collocated samples — two or more portions collected at the same point in time and space so as to be considered identical. These samples are also known as field replicates and should be identified as such. (12)

completeness — evaluates whether all data necessary to perform validation analysis were collected.

compliance — the extent that adherence to SOPs, QAPP, and/or contractual requirements were followed, achieved, and/or completed successfully, and that conditions under which the data were recorded also met the requirements.

1 **conformance** — an affirmative indication or judgment that a product or service has met the
2 requirements of the relevant specification, contract, or regulation; also, the state of meeting the
3 requirements. (1,9,12)

4 **consistency** — determining whether performing data collection procedures across different
5 collection sites (if applicable) and of data reported in multiple places were done in a similar
6 manner.

7 **continuing calibration blank (CCB)** — a blank (zero) standard that is analyzed at a specified
8 rate (e.g., every ten samples) to detect any carryover contamination or instrument drift. (11)

9 **continuing calibration verification (CCV)** — a calibration standard that is analyzed at a
10 specified rate (e.g., every ten samples) to verify instrument stability and performance. (11)

11 **contractor** — any organization or individual contracting to furnish services or items or to
12 perform work. (12)

13 **control limits (upper and lower)** — in statistical quality control, the limits of acceptability
14 shown on control charts; regions outside control limits are defective. (13)

15 **corrective action** — any measures taken to rectify conditions adverse to quality and, where
16 possible, to preclude their recurrence. (9,12)

17 **correctness** — a mechanical, objective check that data collection plans and protocols have been
18 followed and that basic operations and calculations were performed using properly-documented
19 and correctly-applied algorithms.

20 **custodians** — one entrusted with guarding and keeping property, samples or records. (8)

21 **data usability** — the process of ensuring or determining whether the quality of the data produced
22 meets the intended use of the data. (4,12)

23 **data quality** — the appropriate measurement, collection and use of data. (11)

24 **Data Quality Assessment (DQA)** — the scientific and statistical evaluation of data to determine
25 if data obtained from environmental operations are of the right type, quality, and quantity to
26 support their intended use. The five steps of the DQA Process include: 1) reviewing the DQOs
27 and sampling design, 2) conducting a preliminary data review, 3) selecting the statistical test, 4)
28 verifying the assumptions of the statistical test, and 5) drawing conclusions from the data. (12)

Data Quality Objectives Process (DQO Process) — a systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use. The key elements of the process include:

- Concisely defining the problem,
- Identifying the decision to be made,
- Identifying the key inputs to that decision,
- Defining the boundaries of the project,
- Developing the decision rule,
- Specifying tolerable limits on potential decision errors, and
- Selecting the most resource efficient data collection design.

data quality objectives (DQOs) — qualitative and quantitative statements derived from the DQO process that clarify the study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. (5,7,11) Data quality objectives are the qualitative and quantitative outputs from the DQO process. The DQO process was developed originally by the U.S. Environmental Protection Agency, but has been adapted for use by other organizations to meet their specific planning requirements. (5,7)

data reduction — the process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collating them into a more useful form. Data reduction is irreversible and generally results in a reduced data set and an associated loss of detail. (12)

design — the specifications, drawings, design criteria, and performance requirements. Also, the result of deliberate planning, analysis, mathematical manipulations, and design processes. (4, 12)

detection limit (DL) — the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated level of probability. DLs are analyte- and matrix-specific and may be laboratory-dependent. (12)

dilution water — water that has been generated by any method which would achieve performance specifications for ASTM Type II water. (11)

distribution — 1) the appointment of an environmental contaminant at a point over time, over an area, or within a volume; 2) a probability function (density function, mass function, or distribution function) used to describe a set of observations (statistical sample) or a population from which the observations are generated. (12)

document — the physical act of producing any written or pictorial information describing, defining, specifying, reporting, or certifying activities, requirements, procedures, or results. (3)

duplicate samples — two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method, including sampling and analysis. See also collocated sample. (12) Duplicate samples may also be generated in the lab instead of collected in the field. (11)

entity — that which can be individually described and considered, such as a process, product, item, organizations, or combination thereof. (9)

environmental programs — an all-inclusive term pertaining to any work or activities involving the environment, including but not limited to: characterization of environmental processes and conditions; environmental monitoring; environmental research and development; the design, construction, and operation of environmental technologies; and laboratory operations on environmental samples. (12)

environmental data operations — any work performed to obtain, use, or report information pertaining to environmental processes and conditions. (5, 12)

environmental data — any parameters or pieces of information collected or produced from measurements, analyses, or models of environmental processes, conditions, and effects of pollutants on human health and the ecology, including results from laboratory analyses or from experimental systems representing such processes and conditions. (12)

environmental programs — a term pertaining to any work or activities involving the environment, including: characterization of environmental processes and conditions; environmental monitoring; environmental research and development; the design, construction, and operation of environmental technologies; and laboratory operations on environmental samples. (7) Any measurements or information that describe environmental processes or conditions, or the performance of environmental technology, (5)

environmental processes — manufactured or natural processes that produce discharges to or that impact the ambient environment. (5)

estimate — a characteristic from the sample from which inferences on parameters can be made. (12)

field (matrix) spike — a sample prepared at the sampling point (i.e., in the field) by adding a known mass of the target analyte to a specified amount of the sample. Field matrix spikes are used, for example, to determine the effect of the sample preservation, shipment, storage, and preparation on analyte recovery efficiency (the analytical bias). (12)

field blank — a sample used to provide information about contaminants that may be introduced during sample collection, storage, and transport; a clean sample, carried to the sampling site,

exposed to sampling conditions, returned to the laboratory, and treated as an environmental sample. (12)

finding — an assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative, and is normally accompanied by specific examples of the observed condition. (7)

food lot — the processing number assigned to animal food (such as *Artemia* cysts) by the supplier. If no lot number is assigned by the supplier for a food lot then the laboratory will establish a method to assign lot numbers to food materials that are used in animal culturing and testing. (11)

guidance — a suggested practice that is not mandatory, intended as an aid or example in complying with a standard or requirement. (1,12)

holding time — the period of time a sample may be stored prior to its required analysis. While exceeding the holding time does not necessarily negate the veracity of analytical results, it causes the qualifying or ‘flagging’ of any data not meeting all of the specified acceptance criteria. (12) Holding time can also refer to the amount of time a sample was actually stored, whether it meets the designated threshold value for holding or surpasses it. (11)

initial calibration verification (ICV) — the first blank (zero) standard analyzed to confirm the initial instrument calibration. (11)

initial calibration blank (ICB) — the first blank (zero) standard analyzed to confirm the initial instrument calibration. (11)

inspection — an activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (8)

integrity — an unimpaired condition or soundness. A quality or state of being complete or undivided. (8)

item — An all-inclusive term used in place of the following: appurtenance, facility, sample, assembly, component, equipment, material, module, part, product, structure, subassembly, subsystem, system, unit, documented concepts, or data. (4)

level of confidence — the probability associated with a confidence interval; the probability that the interval contains a given parameter or characteristic. (13)

management — those individuals directly responsible and accountable for planning, implementing, and assessing work. (4)

1 **management system** — a structured non-technical system describing the policies, objectives,
2 principles, organizational authority, responsibilities, accountability, and implementation plan of an
3 organization for conducting work and producing items and services. (7)

4 **matrix spike** — a sample prepared by adding a known mass or volume of a target analyte to a
5 specified amount of matrix sample for which an independent estimate of the target analyte
6 concentration is available. Spike samples are used, for example, to determine the effect of the
7 matrix on a method's recovery efficiency. (12)

8 **may** — permission but not a requirement. (4)

9 **memory effects** — the effect that a relatively high concentration sample has on the measurement
10 of a lower concentration sample of the same analyte when the higher concentration sample
11 precedes the lower concentration sample in the same analytical instrument. (12)

12 **method** — a body of procedures and techniques for performing an activity (e.g., sampling,
13 chemical analysis, quantification), systematically presented in the order in which they are to be
14 executed. (12)

15 **method blank** — a sample prepared to represent the sample matrix as closely as possible and
16 analyzed exactly like the calibration standards, samples, and quality control (QC) samples.
17 Results of method blanks provide an estimate of the within-batch variability of the blank response
18 and an indication of bias introduced by the preparation and analytical procedure. (12)

19
20 **method detection limits (MDL)** — the minimum concentration of a substance that can be
21 measured and reported with 99% confidence that the analyte concentration is greater than zero
22 and is determined from analysis of a sample in a given matrix containing the analyte. (11)

23 **method** — a body of procedures and techniques for performing an activity (e.g., sampling,
24 chemical analysis, quantification) systematically presented in the order in which they are to be
25 executed. (7)

26 **mid-range check** — a standard used to establish whether the middle of a measurement method's
27 calibrated range is still within specifications. (12)

28 **must** — a requirement that has to be met. (8)

29 **objective evidence** — any documented statement of fact, other information, or record, either
30 quantitative or qualitative, pertaining to the quality of an item or activity, based on observations,
31 measurements, or tests which can be verified. (1,9)

observation — an assessment conclusion that identifies a condition (either positive or negative) which does not represent a significant impact on an item or activity. An observation may identify a condition which does not yet cause a degradation of quality. (7)

organization — a company, corporation, firm, enterprise, or institution, or part thereof, whether incorporated or not, public or private, that has its own functions and administration. (9)

outlier — an extreme observation that is shown to have a low probability of belonging to a specified data population. (12)

parameter — a quantity, usually unknown, such as a mean or a standard deviation characterizing a population. Commonly misused for "variable," "characteristic," or "property." (12)

performance criteria -- criteria that define the quality of the data relative to the limits of uncertainty allowed for a measurement (bias, precision, minimum detection).

peer review — a documented critical review of work generally beyond the state of the art or characterized by the existence of potential uncertainty. The peer review is conducted by qualified individuals (or organization) who are independent of those who performed the work, but are collectively equivalent in technical expertise (i.e., peers) to those who performed the original work. The peer review is conducted to ensure that activities are technically adequate, competently performed, properly documented, and satisfy established technical and quality requirements. The peer review is an in-depth assessment of the assumptions, calculations, extrapolations, alternate interpretations, methodology, acceptance criteria, and conclusions pertaining to specific work and of the documentation that supports them. Peer reviews provide an evaluation of a subject where quantitative methods of analysis or measures of success are unavailable or undefined, such as in research and development. (4)

precision — a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, expressed generally in terms of the standard deviation. (4,7)

preparation blank (PB) — a laboratory quality control sample which consists of analyte-free matrix processed through the appropriate sample preparation and analysis procedure to document that contaminants are not being introduced to the analytical system during preparation. (11)

probit analysis — a procedure used in dosage-response studies to avoid obtaining negative response values to certain dosages; five is added to the values of the standardized variate which is assumed to be normal; the term is a contraction or probability unit. (13)

procedure — a specified way to perform an activity. (9)

process — a set of interrelated resources and activities that transforms inputs into outputs. Examples of processes include analysis, design, data collection, operation, fabrication, and calculation. (9)

project — an organized set of activities within a program. Specifically, activity in which measurements are used to further a goal. (4,7,11)

quality control (QC) — the overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality. (4,9)

quality system — structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items) and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (2,7,9)

quality management — that aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and the systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality system. (2)

quality assurance (QA) — an integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer. (7)

Quality Assurance Project Plan (QAPP) — a formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria. (4)

quality control (QC) sample — an uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. Generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. (12)

quality — the totality of features and characteristics of a product or service that bear on its ability to meet the stated or implied needs and expectations of the user. (2,7,9)

raw data package — a collection of data that has not been processed. (13)

reagent — a substance used (as in detecting or measuring a component, in preparing a product or in developing photographs) because of its chemical or biological activity. (8)

record (quality) — a document that furnishes objective evidence of the quality of items or activities and that has been verified and authenticated as technically complete and correct. Records may include photographs, drawings, magnetic tape, and other data recording media. (4,9)

recovery — the act of determining whether or not the methodology measures all of the analyte contained in a sample. (12)

requirement — a formal statement of a need and the expected manner in which it is to be met. (12)

research (basic) — a process, the objective of which is to gain fuller knowledge or understanding of the fundamental aspects of phenomena and of observable facts without specific applications toward processes or products in mind. (6,7)

research (applied) — a process, the objective of which is to gain knowledge or understanding necessary for determining the means by which a recognized and specific need may be met. (6,7)

sample custody — the method by which samples are tracked from collection to shipment or receipt by the laboratory. (11)

sensitivity — the capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest. (12)

span check — a standard used to establish that a measurement method is not deviating from its calibrated range. (12)

spatial — relating to, occupying, or having the character of space. (8)

specification — a document stating requirements and which refers to or includes drawings or other relevant documents. Specifications should indicate the means and the criteria for determining conformance. (9)

spike — a substance that is added to an environmental sample to increase the concentration of target analytes by known amounts; used to assess measurement accuracy (spike recovery). Spike duplicates are used to assess measurement precision in cases where actual samples are not or cannot be duplicated. (11, 12)

standard deviation — a measure of the dispersion or imprecision of a sample or population distribution expressed as the positive square root of the variance and has the same unit of measurement as the mean. (12)

standard operating procedure (SOP) — a written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps, and that is officially approved as the method for performing certain routine or repetitive tasks. (4)

surrogate percent recoveries — the actual amount of surrogate analyte recovered as a result of the analysis divided by the known amount of surrogate introduced to the analytical system multiplied by 100. (11)

surrogate — an organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. (11)

surrogate spike or analyte — a pure substance with properties that mimic an analyte of interest. It is unlikely to be found in environmental samples and is added to them to establish that the analytical method has been performed properly. (12)

systematic planning -- a process that is based on the scientific method and uses a graded approach to ensure that the level of detail in planning is commensurate with the importance and intended use of the work and available resources.

task — defined or specified portion of a project. (11)

technical usability — a technical assessment of an overall data set based on the results and the related quality control data to determine if the data “make sense” and are fit for use. (11)

technical systems audit (TSA) — a thorough, systematic, on-site qualitative audit of facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a system. (12)

temporal — of or relating to time. (8)

traceability — the ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project work to the requirements for quality for the project. (9, 12)

type — a particular kind, class or group. (8)

usability — capable of being used; convenient and practical for use. (8)

user — when used in the context of environmental programs an organization, group, or individual that utilizes the results or products from environmental programs. A user may also be the customer for whom the results or products were collected or created. (7)

validation — confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. In design and development, validation concerns the process of examining a product or result to determine conformation to user needs. (9)

variance (statistical) — a measure or dispersion of a sample or population distribution. (12)

verification — confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. In design and development, validation concerns the process of examining a result of a given activity to determine conformance to the stated requirements for that activity. (9)

LIST OF SOURCES FOR DEFINITIONS

- (1) ANSI/ASQC A3-1987, *Quality Systems Terminology*.
- (2) ANSI/ASQC Q9004-1-1994, *Quality Management and Quality System Elements – Guidelines*.
- (3) ASME NQA-1, *Quality Assurance Program Requirements for Nuclear Facilities*, 1989 edition.
- (4) *TRADE Quality Assurance Resources Guide*, “Glossary of Terms, Abbreviations, and Acronyms,” Training Resources and Data Exchange (TRADE), Oak Ridge Institute for Science and Education (July 1991).
- (5) U.S. EPA, *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* (QAMS-005/80), December 29, 1980.
- (6) U.S. DOE Order 5700.6C, *Quality Assurance*.
- (7) ANSI/ASQCE4-1994, *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*.
- (8) *The Random House College Dictionary*.
- (9) ISO 8402:1994, *Quality Management and Quality Assurance – Vocabulary*.
- (10) *Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, Eighth Edition (March, 1991).
- (11) Consensus definition from multiple sources and reviewer comments.
- (12) *EPA Guidance For Quality Assurance Project Plans (QA/G-5)*.
- (13) *McGraw Hill Dictionary of Scientific and Technological Terms*, 5th Edition.

(This page left blank intentionally.)

APPENDIX B

DESCRIPTION OF RESPONSIBILITIES FOR JOBS DETAILED IN TABLES 3-1 THROUGH 3-4

The job descriptions provided below serve as examples of job tasks which key staff members would perform as part of an environmental data operation. The lists are not exhaustive and therefore do not necessarily contain every task that may be required of a particular person in the course of a project. In turn, all data operations may not require all of these tasks, and certain staff may perform multiple job descriptions.

Figure B-1 provides an example of an organizational chart showing the interaction and reporting responsibilities of the key staff on an environmental data operation. This chart shows how the organization charged with doing an environmental project reports to the sponsor or regulatory agency that mandates the work. The organization typically assigns a project manager to the task, who has a number of key personnel reports (e.g., leaders of field, laboratory, and database efforts). The number and responsibilities of key personnel will differ from project to project (e.g., some projects may not have field activities and therefore would not have field personnel, some projects may require multiple laboratory managers if multiple laboratories are to be used, certain research projects may require a project statistician reporting to the project manager).

An important point that is conveyed in Figure B-1 is that the QA Administrator reports to someone in the organization other than the project manager. This is done to ensure that the QA Administrator can execute his/her project responsibilities in an objective manner without the potential for outside influence from the project manager or other project staff. The QA Administrator is responsible, however, for providing an open line of communication with the project manager on the results of QA audits and other investigations, as seen by the dotted line in Figure B-1.

Project Manager:

- Assure necessary training and certification of staff.
- Document staff skills and ability to perform the required work.
- Assign field and laboratory custodians and perform a review of their responsibilities.
- Ensure necessary communication between project staff members and with the remainder of the organization.
- Act upon information provided by the QA Administrator.

QA Administrator:

- Maintain QA and QC of laboratory practices, field practices, and other standard procedures.

- Document corrective actions taken in field and laboratory when violations occur.
- Document general QC measures and results.
- Thoroughly document the chain of custody issues such as log-in, sample integrity, transfer among contractors and necessary storage of sample.
- Verify that proper sample identification has occurred.
- Verify the preparation data and time of analytical standards.
- Verify any corrective action taken if calibration or calibration check data fail to meet the acceptance criteria.
- Oversee any necessary QC checks of data entry.

QA Auditor:

- Verify the preparation data and time of analytical standards.
- Plan and perform audits.
- Verify corrective actions.

Field Team Leader:

- Verify use of proper sampling protocols in the field.
- Act as a project liaison for all field activities.
- Verify that appropriate QA and QC field activities have been performed and that appropriate corrective action is taken.

Field Technical Personnel:

- Maintain and document chain of custody of samples throughout life of sample.
- Document general QC measures and results.
- Inspect and maintain field equipment.
- Maintain an accurate written record of sample handling from collection through disposal.
- Perform and document all necessary instrument maintenance and inspections.

Sample Custodian:

- Maintain and document chain of custody of sample throughout the life of the sample, including any problems that may arise at any point from initial collection through final disposal.
- Verify sample integrity and proper handling and receipt.
- Continue to thoroughly document the chain of custody issues such as log-in, sample integrity, transfer among contractors and necessary storage of samples.

1

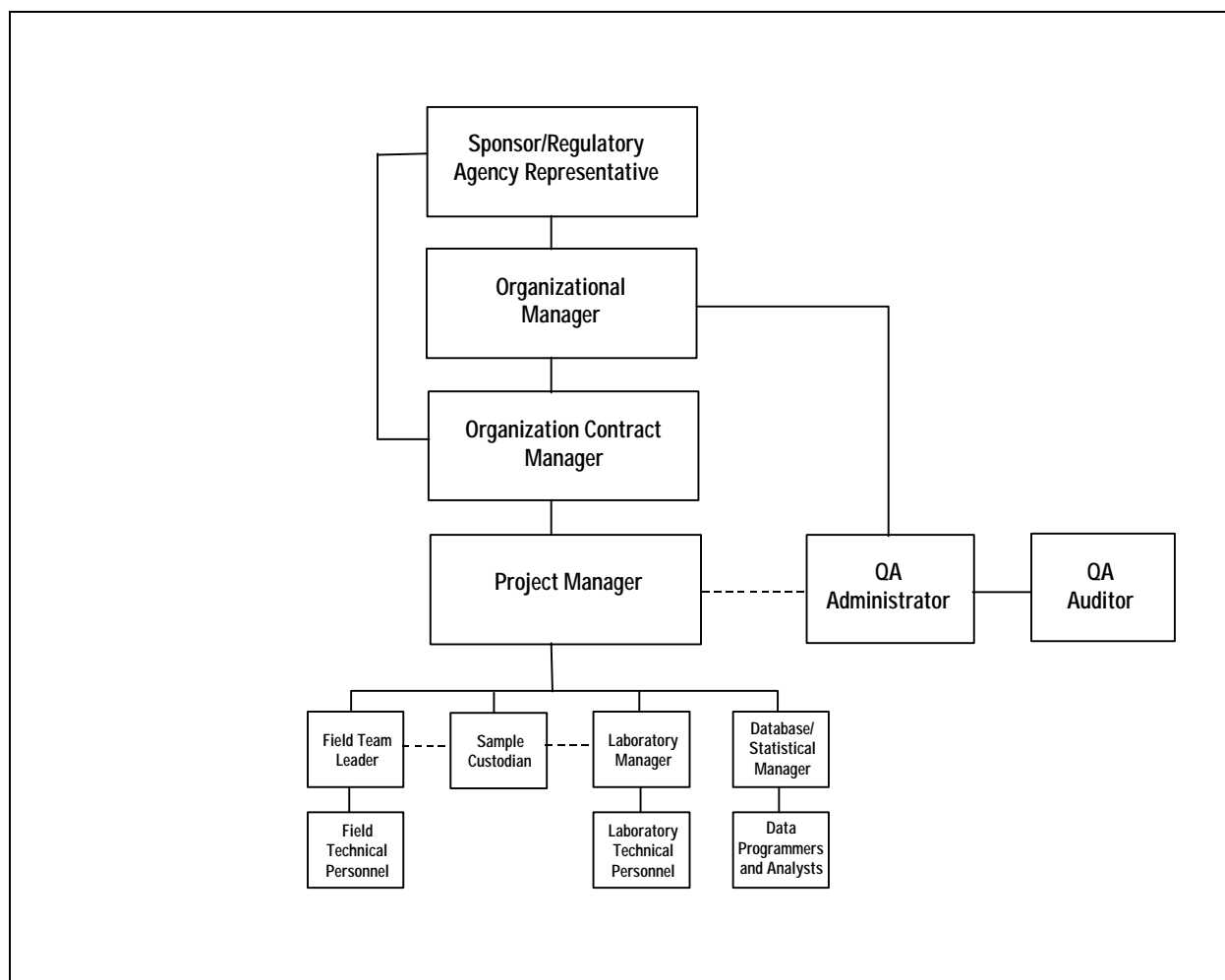


Figure B-1. Example of an Organizational Chart for an Environmental Data Operation

2

Laboratory Manager:

- 3 • Identify and document appropriate analytical protocols for the project.
- 4 • Ensure that necessary laboratory resources are available for the project. Work with
- 5 technical personnel to ensure that their tasks (detailed below) are properly performed and
- 6 completed.
- 7 • Act upon necessary corrective action to be taken by the laboratory.

Laboratory Technical Personnel:

9

- 10 • Maintain and document chain of custody of samples throughout life of sample.
- 11 • Maintain QA and QC of measurements system.
- 12 • Document general QC measures and results.
- 13 • Inspect and maintain laboratory equipment.

- 1 • Maintain laboratory analysis records by overseeing use of proper analytical methods and
- 2 documenting failures of analytical system.
- 3 • Maintain an accurate written record of sample handling from collection through disposal.
- 4 • Confirm calibration of instruments against accepted standards.
- 5 • Verify that the Continuing Calibration Blank was analyzed using proper settings.
- 6 • Monitor performance of instruments by continually checking calibration.
- 7 • Verify analysis of Initial Calibration Blank after calibration standards and test that it is
- 8 within acceptable limits.
- 9 • Perform instrument calibration for accurate establishment of relationship between actual
- 10 sample concentration and instrument response.
- 11 • Perform and document all necessary instrument maintenance and inspections to maintain
- 12 proper working order.
- 13 • Determine the laboratory's inter-element and background correction by performing the
- 14 Interference Check Sample.
- 15 • Analyze Laboratory Control Sample under strictest of conditions and verify that results
- 16 are within acceptable limits.
- 17 • Prepare and analyze at least one Preparation Blank.
- 18 • Ensure that requirements are met to provide reproducible reference point in traceability
- 19 standards review.

20 Database/Statistical Manager:

- 21 • Ensure that necessary database and computer resources are available for the project.
- 22 • Oversee the statistical design of sample collection, if such a design is warranted, and
- 23 evaluate the extent to which this design was followed and the effects of any deviation from
- 24 the design.
- 25 • Establish statistical analysis objectives, if statistical analysis is to be performed
- 26 • Identify and obtain appropriate database and statistical software necessary to manage the
- 27 collected data.
- 28 • Establish the procedure for data transfer from the laboratory to the data bases.
- 29 • Coordinate appropriate QC checks on the data with the QA Administrator.

30 Data Programmers and Analysts:

- 31 • Properly document all data programs, spreadsheets, and data bases.
- 32 • Include automated data checks within the data base to minimize the likelihood of invalid
- 33 data being stored in the database.

APPENDIX C

ADDITIONAL CASE STUDIES

This appendix will introduce two case studies that are included in this guidance to help illustrate how data verification and validation procedures are applied in real-world environmental studies (both in the field and in the laboratory). To supplement the field sampling of chemical data case study that was presented throughout body of the paper and to cover the range of environmental data that might be collected to support EPA regulatory and research objectives, one case study focuses on laboratory study of chemical data, and the second on biological data. The scopes of these studies have been modified as necessary to suit the purposes of the guidance. The objective of including the case studies is to allow the user to better understand the definitions of data verification and data validation and which verification and validation procedures are important to implement in various situations. The case studies will provide additional insight on the specific examples of verification and validation procedures cited in Chapter 2 and will illustrate their application.

CASE STUDY C.1: CHARACTERIZING HUMAN EXPOSURE TO METALS

Project Description: This case study represents a study that generates analytical data within a chemistry laboratory. It is based on the National Human Exposure Assessment Survey (NHEXAS), in which environmental samples were collected from approximately 450 homes and analyzed for target analytes in order to determine the distribution of total human exposure to metals. Multiple pathways of exposure included ingestion, inhalation, and dermal absorption. Among the target analytes were metals, representing one of the major classes of environmental contaminants. Over a 2-year period, samples of air, dust, and soil were collected in this study and

- perform evaluations of relationships between exposure reports, environmental measurements, and biomarkers of target pollutants;
- predict total exposures;
- assess total exposures in minority and disadvantaged subsets of the population.

The data will be collected in three stages. In stage one, approximately 1200 households will be interviewed using NHEXAS questionnaires. In stage two, environmental sampling will take place in approximately 450 households representatively selected from respondents. Environmental sampling will include metals in dust, soil, outside air, and tap water. Stage three will be a reevaluation for metals in a subset of representative households using methods with greater resolution. Subjects in the households will provide biological samples. The measured parameters will include sociodemographic and residential characteristics, time-activity patterns, exposure factors (e.g., food and water consumption patterns), carrier-medium concentrations (e.g., breathing zone air), and body burden (e.g., in blood and urine).

Survey design calculations are based on the delineation of population subgroups within the design chosen with a power of 0.80 and a type-I error (p -value) of 0.05 for the detection of specific groups and those at the 90th percentile of exposure- $p_1 = 0.50$ and $p_2 = 0.10$ for the final stage. In this survey, the number of samples necessary have been determined, as has the ability to distinguish between upper and mid-range of the exposure distributions.

Data from this study to be verified and validated include

- equipment information (e.g., make model identification, calibration records);
- sample and quality control results (e.g., initial and continuing calibration verification standards; reference material; and laboratory blanks, spikes, and duplicates); and
- sample chain-of-custody documentation.

Data Verification Within Case Study C.1

Table C-1 provides an illustration of how the verification procedures or steps might be implemented in all three case studies introduced earlier. Because this document is intended to be used to provide input to the QAPP, the language in Table C-1 reflects proposed data verification activities. As noted above, not all of the verification procedures are required, so some of the verification steps or procedures presented in Table C-1 are not implemented in one of more of the case studies.

Table C-2 provides an illustration of how the correctness procedures or steps might be implemented in the three case studies. Note that some of the steps or procedures are not implemented in one or more of the case studies.

Table C-3 provides an illustration of how the consistency procedures or steps might be implemented in the three case studies. Note that each step or procedure may not necessarily be performed in each of the case studies.

Table C-4 provides an illustration of how the completeness procedures or steps might be implemented in the three case studies. Note that each step or procedure may not necessarily be performed in each of the case studies.

General responsibilities for the job titles mentioned in Tables C-1 through C-4 are summarized in Appendix B. Note that one staff member may hold multiple job titles.

Table C-1. Examples of How Data Compliance Procedures Can Be Applied Within Case Study C.1

Procedure or Verification Step	Approach Taken in Case Study C.1
Training and Certification of Staff	Laboratory management will be responsible for assigning qualified personnel to each task involved in the preparation and analysis of samples. Laboratory technicians will be trained to follow laboratory SOPs, and this will be documented in a training record containing the date training was performed, the name of the trainee, the task, trainee's level of ability, and name of the instructor. Chemists and analysts will be provided formal training by the ICP/MS instrument manufacturer. Training certificates will become part of the employee's training file.
Assign Sample Custodians	The laboratory manager will assign a Sample Custodian, who will be responsible for meeting all sample handling and COC requirements while the samples are in the possession of the laboratory. This will include documenting sample receipt and COC, locking samples in a designated refrigerator and keeping control of the key, and documenting sample storage and retrieval in the laboratory sample log book. An alternate Sample Custodian will be assigned to perform these duties in the case of absence of the Sample Custodian.
Field Data Collection Audit	Not applicable
Confirm Calibration Verification Calculations	The ICP/MS analyst will be responsible for performing this verification check by recalculating the ICV and CCV percent recovery results manually at a frequency of 10% to ensure that the recalculated value agree with the laboratory reported value. The equation used will be $\% \text{Recovery} = ([\text{found}]/[\text{true}]) \times 100$. Calculation checks will be documented in the instrument calibration logbook.
Method Detection Limits Audit	The ICP/MS analyst will be responsible for verification of MDLs for each analyte of interest and each matrix involved in the study. MDLs will be verified by digesting and analyzing seven replicate samples at a level approximately five times the estimated MDL, calculating the standard deviation and multiplying by the student-t value for 99% confidence.
Blank Sample Collection and Analysis	Laboratory technical personnel will be responsible for the preparation of blanks at the frequency required by the QAPP. Reagent blanks will be prepared by carrying all laboratory reagents through the preparation procedure and analysis. Method blanks will be prepared by digesting blank sample media to assess the response of the laboratory analytical system.

**Table C-1. Examples of How Data Compliance Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
1 2 3 Sample Preservation and Handling	The Laboratory Sample Custodian will be responsible for initial sample preservation upon receipt at the laboratory. Soil samples will require refrigeration during shipment and once received at the laboratory. The Sample Custodian will document whether appropriate refrigeration or cooling is maintained during shipment and then place the samples in the refrigerator and enter that action in the sample database.
4 Sample Storage	The QA Administrator will be responsible for verifying that sample storage requirements and holding times are followed in accordance with EPA Methods. The audit will consist of comparing sample COC and log in documentation with sample preparation SOPs to determine whether samples are digested and analyzed within the appropriate holding times. By reviewing the sample record information that documented the date and time of digestion and sample records containing date and time received, the QA Administrator will determine whether all soil samples are prepared within the appropriate holding times. A review of the refrigerator temperature logs and COC forms that documented sample location (refrigerator) will be used to verify that soil samples have been held at the appropriate temperature. Compliance will be noted in the audit report.
5 6 7 Calibration Check Sample Analysis	See ICV, CCV, CCB
8 9 10 Duplicate Sample Collection and Analysis	The laboratory technician will be responsible for the preparation of laboratory duplicates used to assess the precision of the analytical system for each matrix. Laboratory duplicates will be digested at a frequency of 5% in accordance with the requirements in the QAPP. The ICP/MS analyst will be responsible for the analysis of the laboratory duplicates and analysis of analytical replicates used to assess instrument precision. This will be accomplished by analyzing one sample digestate in duplicate after every tenth sample measurement. The relative percent difference (RPD) control limit for laboratory and analytical duplicates is defined in the QAPP at less than 20%. The calculation for RPD is $\frac{[\text{sample}] - [\text{duplicate}]}{\text{average}([\text{sample}], [\text{duplicate}])} \times 100$. The Laboratory Manager will be responsible for reviewing the raw data to determine that the appropriate number of duplicate samples are prepared and analyzed and that duplicate results for each method and analyte fall within the established control limits. Duplicate samples that fall outside control limits will be re-digested and analyzed or reanalyzed depending upon the type of duplicate sample along with the associated sample batch.
11 Field Blank	Not applicable
12 13 Field Duplicate Collection	Not applicable

**Table C-1. Examples of How Data Compliance Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
1 Calibration	The ICP/MS analyst will be responsible for initial review of the instrument calibration. The calibration audit will confirm proper instrument calibration with a NIST traceable standard from a source independent of the calibration standards (e.g., Standard Reference Material (SRM)). Acceptance limits for percent recovery of SRM are defined in the QAPP at $100 \pm 10\%$. In addition, the linear regression curve will be printed out and the correlation coefficient will be calculated for each analyte of interest. The correlation coefficient (r) will then be compared to the requirement in the instrument SOP. Deviations will require immediate termination of analysis and corrective action.
2 3 4 Calibration Standard Preparation Audit	The QA Auditor will perform this audit by first reviewing the laboratory standard preparation logbook for conformance to SOPs and inspecting calibration standard containers and labels. The QA Auditor will also observed the process of making standards to ensure that laboratory equipment such as Class A pipettes and volumetric flasks are being used properly. Calibration records for previous analyses, including linear regression curves and correlation coefficients will be reviewed, and one standard will be chosen at random to verify its traceability to NIST certified standards.
5 6 7 Interference Check Sample (ICS) Analysis	The ICP/MS analyst will be responsible for analysis of the Interference Check Sample (ICS) used to quantify potential interferences for the analytes of interest. No control limits are specified in the QAPP, so the recovery will be plotted on control charts and monitored over time. If the recovery falls outside the 3σ control limits for the last 35 data points, the analyst will terminate analysis and implement corrective action. This activity will be documented in the ICP/MS calibration logbook and will also be part of the electronic calibration record.
8 9 10 Initial Calibration Blank (ICB) Analysis	The ICP/MS analyst will be responsible for analysis of the Initial Calibration Blank (ICB) used to verify instrument calibration. This activity will be documented in the ICP/MS calibration logbook and will also be part of the electronic calibration record. Analysis of the ICB above the MDL or outside the control limits will be repeated and if confirmed, the analytical system will be recalibrated.
11 12 13 Initial Calibration Verification (ICV) Analysis	The ICP/MS analyst will be responsible for analysis of the Initial Calibration Verification (ICV) sample used to demonstrate that the initial calibration is valid using a mid-range standard made from a source independent of the working standards. The ICV will be analyzed immediately following instrument calibration. Control limits for ICV percent recovery are defined in the QAPP at $100 \pm 10\%$. If recovery falls outside the limits, corrective action including recalibration will be required. These activities will be documented in the ICP/MS calibration logbook and will also be part of the electronic calibration record.
14 15 16 17 Continuing Calibration Verification (CCV) Analysis	The ICP/MS analyst will be responsible for analysis of the Continuing Calibration Verification (CCV) sample used to demonstrate that the initial calibration is still valid by checking the performance of the instrument on a continual basis. A mid-range working standard will be analyzed after each set of ten samples analyzed. Control limits for CCV percent recovery are defined in the QAPP at $100 \pm 10\%$. If recovery falls outside the limits, corrective action including recalibration will be required. These activities will be documented in the ICP/MS calibration logbook and will also be part of the electronic calibration record.

**Table C-1. Examples of How Data Compliance Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
1 Continuing Calibration Blank (CCB) Analysis 2 3	The ICP/MS analyst will be responsible for analysis of the Continuing Calibration Blank (CCB) sample used to demonstrate that the initial calibration is still valid by checking the baseline performance of the instrument on a continual basis. The calibration blank will be analyzed after each set of ten samples analyzed. Control limits for CCB are defined in the QAPP as greater than the MDL for each analyte. If recovery falls outside the limits, corrective action including recalibration will be required. These activities will be documented in the ICP/MS calibration logbook and will also be part of the electronic calibration record.
4 Spike Sample Analysis 5	The laboratory technician will be responsible for the preparation of matrix spikes used to assess the analytical bias for each matrix. Matrix spikes will be prepared at a frequency of 5% in accordance with the requirements in the QAPP. The ICP/MS analyst will be responsible for the analysis of the matrix spikes and the preparation and analysis of analytical spikes used to assess instrument bias. In preparation of the analytical spike, the spike will be added to the sample after digestion. The Laboratory Manager will be responsible for reviewing the raw data to determine that the appropriate number of matrix spikes and analytical spikes are prepared and analyzed and that results for each method and analyte fall within the established control limits. Spiked samples that fall outside control limits will be re-digested and analyzed or reanalyzed along with the associated sample batch depending upon the type of spike.
6 Calibration Corrective Action Audit 7 8	The QA Auditor will be responsible for periodic audits of laboratory calibration records to verify that appropriate corrective action is taken and documented if calibration or calibration check data fail to meet the acceptance criteria in the QAPP. The ICP/MS calibration logbook will be reviewed for consistency with the instrument printouts.
9 Method Blank Analysis 10	Laboratory personnel will be responsible for preparation and analysis of at least one method blank for each matrix. The laboratory manager will coordinate receipt of blank media for each matrix from the sample collection staff. For example, the client will ship blank wipes along with dust wipe samples that are exactly the same material as those used in sample collection. These blank wipes will be prepared and analyzed by the same procedure as the samples.
11 Laboratory Control Sample (LCS) Analysis 12 13	The ICP/MS analyst will be responsible for ensuring that the appropriate number of LCS are digested and analyzed and the results are within acceptable control limits. The percent recovery limits that are established in the program QAPP for LCS are $100 \pm 10\%$.

**Table C-2. Examples of How Data Correctness Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
Instrument Inspection and Maintenance Audit	The QA Auditor will be responsible for verifying that the procedures for laboratory instrument inspection and maintenance are followed. The ICP/MS inspection and maintenance logbook will be reviewed along with all applicable SOPs and the instrument manual.
Instrument Calibration Review	The ICP/MS analyst will be responsible for performing the ICP/MS calibration review. The daily review will be performed to ensure that calibration is performed within an acceptable time prior to the generation of measurement data and in proper sequence and to verify that calibration is performed using standards that bracket the range of reported measurement results. Records that will be reviewed include standard preparation logbooks, instrument logbooks, instrument printouts, and raw data.
Data Recording Audit	The QA Auditor will perform data recording audits periodically throughout the study. Raw data and sample preparation logbooks will be reviewed for compliance to data recording procedures.
Data Reduction Audit	The QA Auditor will be responsible for verifying the data reduction process. Raw data will be entered into a database both manually and electronically. MS Excel macros will be used to summarize sample concentration data (in $\mu\text{g/L}$) into tables, and it will be the responsibility of the QA Auditor to verify that the data are properly transformed into reportable data. The QA Auditor will review the raw data for soil samples, including dry weights in grams (g), final sample volume (mL), and sample concentration ($\mu\text{g/L}$) and recalculate the final concentration in mg/kg (or, equivalently, $\mu\text{g/g}$) for several samples to ensure the data reduction program is working properly.
Data Transformation Audit	The ICP/MS analyst will be responsible for review of the electronic data transformation performed by the ICP/MS report program. Analyte measurement intensities will be electronically transformed into concentrations ($\mu\text{g/L}$) by the instrument software using a simple linear regression technique ($y = mx + b$). The analyst will verify that intensities are being properly transformed into $\mu\text{g/L}$ by entering the calibration standard intensities into a spreadsheet and performing linear regression calculations. Sample intensities will then be used to calculate sample concentration and compared to instrument printouts.
Raw Data Audit	The QA Auditor will be responsible for review of the raw data. This audit will include review of the instrument printouts that contain raw data from the instrument and sample preparation logbooks that contain sample weights, final volumes, and other preparation raw data. These records will be examined to verify that the laboratory reports correct calculations of the sample results.

**Table C-3. Examples of How Data Consistency Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
Data Handling Audit	The ICP/MS analyst will be responsible for checking 10% of all computer calculations manually and documenting these checks in the analysis logbook. The QA Auditor will be responsible for reviewing the logbook to ensure that this verification procedure is performed at the specified frequency using the correct equations.
Data Transmittal Review	The Laboratory Manager will be responsible for the review of the data transfer steps to ensure a minimal loss of data during transmittal. All ICP/MS raw data will be electronically transferred to the report output directory on the network as a read-only file. Laboratory personnel can then access the raw data from any computer workstation and transmit the data electronically to the database manager after review. The data transmittal audit will consist of tracing the path of raw data through this transmittal process to ensure that data arrive in the database without transmittal errors.

**Table C-4. Examples of How Data Completeness Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
Documentation of Field Corrective Action	Not applicable
Sample Records Documentation and Audit	The Sample Custodian will be responsible for initial receipt of samples in the laboratory and documentation of sample handling and COC. The Custodian will note sample temperature, condition, and preservation on the COC forms. Once sample custody is relinquished to laboratory personnel, the COC forms will be placed in a three-ring binder in the laboratory, and laboratory personnel will be responsible for maintaining all sample records using controlled logbooks. The laboratory technician will log samples into the logbook and enter sample preparation information such as sample weights and volumes prior to digestion. QC sample information, such as laboratory duplicate sample identification, will be recorded there also. Method used for sample preparation, technician initials, and date will be required entries. Sample records for analysis will be computer records generated by the ICP/MS that contained the date of analysis, method used, analyst initials, sample identification, and analyte concentration results. In addition, the analyst will be required to document any information that could impact the quality of the results in a controlled bound laboratory logbook, which will be issued, tracked, and reviewed by the QA Auditor.
Sample Shipment Documentation and Audit	Not applicable

**Table C-4. Examples of How Data Completeness Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
1 2 3 Data Management Audit	The QA Auditor will perform a data management review prior to generation of the final report to the client. This will be performed to ensure that data generated in the laboratory can be traced from receipt and log in through sample archive and raw data storage. The bar code system will facilitate data management throughout the study, and the QA Auditor will be able to trace the path of data without finding system failures.
4 5 Chain of Custody Documentation	Upon receipt at the laboratory, the Laboratory Sample Custodian will be responsible for receiving the samples and documenting all required information on the COC form. The sample custodian will first verified that all samples are actually received, in good condition, and at the proper temperature. Any discrepancies will be documented on the COC form. Each sample will then assigned a bar code, which will be affixed to the sample container and its corresponding COC form. Using a bar code scanner, samples will then be logged in electronically and this information will be entered into a database. Once the Sample Custodian either places the samples in storage or relinquishes them to laboratory personnel, the original COC forms will be filed in three ring binders in the laboratory.
6 Sample Log In	The Laboratory Sample Custodian will log in samples upon receipt at the laboratory. Documentation for log-in will include sample custodian name, date, time, condition of samples, preservation, and any violations to sample handling procedures that occurred. This information will be entered electronically into a database along with sample identification information, which was entered using a bar code system. Each sample will be issued a unique bar code that will be used to track the sample location throughout the study.
7 8 9 Sample Identification Audit	The QA Auditor will be responsible for ensuring that sample identification procedures are maintained throughout all phases of laboratory sample preparation and analysis. The QA Auditor will review procedures for tracing sample IDs from the original sample containers to laboratory beakers, volumetric flasks, and instrument auto-sampler tubes.
10 11 12 13 Instrument Inspection and Maintenance Documentation	Laboratory personnel will be responsible for inspection and maintenance of laboratory instruments. Manufacturers' instrument manuals and laboratory SOPs will define the specific maintenance activities that are required and at what frequency they must be performed. The ICP/MS maintenance activities that are part of the daily optimization will be documented in the ICP/MS inspection and maintenance logbook. All routine activities as well as problems will be written in the logbook. It will be the responsibility of the analyst to identify corrective action and document it.
14 15 Traceability of Standards Review	Laboratory personnel will be responsible for documenting appropriate information to ensure traceability of instrument calibration standards. All stock standards that are purchased for the study will be certified NIST traceable and will be accompanied by a certificate of analysis that will be kept on file in the laboratory. Laboratory technicians will prepare working standards and document the stock standard lot number and date of preparation in the lab standard preparation logbook along with date of expiration and initials. The working standards will be labeled with the following information: date made, concentration, lot number, analyte, <i>prepared by</i> initials, and date of expiration.

**Table C-4. Examples of How Data Completeness Procedures Can Be Applied
Within Case Study C.1**

Procedure or Verification Step	Approach Taken in Case Study C.1
Documentation of Calibration Corrective Action	The ICP/MS analyst will be responsible for the documentation of corrective action taken to resolve problems during calibration of the instrument. Instrument printouts will contain hand-written notes describing correction activities and reasons for performing them. The calibration logbook will contain detailed information for each failed optimization and calibration check. Out of control points on the control charts will be circled and a description of corrective action will be required for any such point.
Laboratory Analysis Records Review	The Laboratory Manager will be responsible for ensuring that the appropriate preparation and analytical method is used for each batch of samples received at the laboratory, that laboratory SOPs are followed, and that any failure in the analytical system is properly documented and corrected.
Documentation of QC Results including Control Charts	The ICP/MS analyst will be responsible for generating the quality control data used to demonstrate instrument capability, calibration, calibration verification, and analytical performance. These results will be documented in the form of instrument printouts as part of the raw data package. Quality control checks will be electronically entered onto control charts in real time and the analyst will be responsible for initial review of the QC check results. The QA Auditor will be responsible for review of these results and their corresponding control charts.
Documentation of Laboratory Corrective Action	Laboratory personnel will be responsible for documentation of all corrective action procedures identified in cases where standard laboratory procedures are violated.

Data Validation Performed Within Case Study C.1

Data validation for Case Study C.1 involves review of the laboratory documentation to ensure that the requirements stated in the QAPP have been met. The validation for this laboratory study considers whether the data collected can be used to evaluate human exposure to metals by various pathways.

The most critical element of the laboratory study is precision and accuracy of the analytical data. The data validation process includes a review of laboratory quality control data to ensure that requirements for precision and accuracy defined in the QAPP have been met. Measurement bias and measurement error outside the limits defined in the study design will have an impact on the agency's ability to use the data to assess human exposure.

The completeness of the data will also impact the usability. The objective for data completeness is defined as 80%. However, the study is designed to evaluate exposure to metals based on location (rural, urban) by sampling 500 homes in three regions. If 20% of the data are lost, and the lost data represents one region, the data can not be used to compare this area to the others. Or if all wipe samples are lost, the dermal absorption pathway can not be properly evaluated.

1 **Case Study C.2: TOXICITY TESTING OF POTW EFFLUENT USING *MYSIDOPSIS***
2 ***BAHIA***

3 Project Description: In this case study, which addresses the reporting of biological data,
4 the toxicity of water collected from a publicly owned treatment works (POTW) was evaluated by
5 conducting a 96-hour static-renewal toxicity test on the sample. In the test, laboratory-reared 24-
6 hour old *Mysidopsis bahia* were used as the test organism. The toxicity test was conducted
7 according to USEPA (1993), as required by the POTW's discharge permit. The 96-hour test
8 consisted of 5 dilutions of effluent, a dilution water control, and a copper sulfate reference
9 standard. Required test conditions were maintained throughout the test. At the end of the test, a
10 probit analysis was performed on the data to determine the estimated LC50 (i.e., the effluent
11 percentage associated with a 50% average survival).
12

Data Quality Objectives

- sample chain of custody documentation.

Data Verification Within Case Study C.2

Table C-5 provides an illustration of how the verification procedures or steps might be implemented in all three case studies introduced earlier. Because this document is intended to be used to provide input to the QAPP, the language in Table C-5 reflects proposed data verification activities. As noted above, not all of the verification procedures are required, so some of the verification steps or procedures presented in Table C-5 are not implemented in one or more of the case studies.

Table C-6 provides an illustration of how the correctness procedures or steps might be implemented in the three case studies. Note that some of the steps or procedures are not implemented in one or more of the case studies.

Table C-7 provides an illustration of how the consistency procedures or steps might be implemented in the three case studies. Note that each step or procedure may not necessarily be performed in each of the case studies.

Table C-8 provides an illustration of how the completeness procedures or steps might be implemented in the three case studies. Note that each step or procedure may not necessarily be performed in each of the case studies.

General responsibilities for the job titles mentioned in Tables C-5 through C-8 are summarized in Appendix B. Note that one staff member may hold multiple job titles.

**Table C-5. Examples of How Data Compliance Procedures Can Be Applied
Within Case Study C.2**

Procedure or Verification Step	Approach Taken in Case Study C.2
Training and Certification of Staff	It will be the responsibility of the Project Manager to ensure that staff are adequately trained. Training at the laboratory will be documented for each SOP procedure that staff can perform. The QA office will provide the Project Manager with a list of staff who need updated training when an SOP is updated.
Assign Sample Custodians	The Project Manager will be responsible for assigning the sample custodian role. The assignment will include a review of qualifications and training to ensure that the designated staff member has specific training in sample receipt, handling, and chain-of-custody procedures. The sample custodian position may be shared by several staff who also had other responsibilities.
Field Data Collection Audit	Not applicable

**Table C-5. Examples of How Data Compliance Procedures Can Be Applied
Within Case Study C.2**

Procedure or Verification Step	Approach Taken in Case Study C.2
Confirm Calibration Verification Calculations	Not applicable
Method Detection Limits Audit	The QA Auditor will be responsible for assessing the sensitivity of the test system. (Method Detection Limits do not apply to toxicity testing). The results of ongoing reference tests will be reviewed to confirm that the test organism sensitivity is constant over time and that reference test LC50s are within the control chart control limits.
Blank Sample Collection and Analysis	Not applicable
Sample Preservation and Handling	The POTW operator will be responsible for initial sample preservation (4°C). The samples will be packed in a cooler with ice. The sample custodian will be responsible for documenting sample preservation and for storing the sample at 4°C. The laboratory technical personnel will be responsible for maintaining the sample at this temperature during the test when the sample is used for daily renewals. The sample will be stored in custody or laboratory refrigerator upon receipt and during testing, respectively. The temperatures of these units will be monitored and documented daily.
Sample Storage	The QA Auditor will be responsible for performing the sample storage audit. The audit will consist of a review of the following records: COC, sample receipt, thermometer calibration, and refrigerator log. The audit will verify that the refrigerator monitoring is performed using a NIST-traceable thermometer which has been calibrated within the year. The appropriate correction factor will be applied. All calibration records and daily monitoring records will be maintained in logbooks.
Calibration Check Sample Analysis	The laboratory technical personnel will be responsible for calibration check sample analysis. Check samples for the pH meter, refractometer, and DO meter will be analyzed according to the SOP requirements: the refractometer "zero" is verified with deionized water at the beginning and end of measurements; the DO Meter range is verified using nitrogen-purged and oxygen saturated seawater; the pH meter calibration is verified using a buffer that approximated the sample pH ensure that there has been no instrument drift.
Duplicate Sample Collection and Analysis	Not applicable
Field Blank	Not applicable
Field Duplicate Collection	Not applicable
Calibration	The laboratory technical personnel will be responsible for performing a calibration review immediately after instrument calibration to ensure that it meets the SOP acceptance criteria. All calibration solutions will be NIST traceable (if available) as will be the balance calibration weights. The calibration requirements for water quality instruments and the balances will be printed directly on the instrument log forms so that a comparison can be made immediately between the SOP requirements and the results of the calibration.

**Table C-5. Examples of How Data Compliance Procedures Can Be Applied
Within Case Study C.2**

Procedure or Verification Step	Approach Taken in Case Study C.2
1 Calibration 2 Standard 3 Preparation Audit	The QA Auditor will be responsible for performing this audit. The audit of pH buffer solution preparation will be performed as an in-phase inspection. During this inspection the buffer salts containers will be examined for proper labeling and will be traced to the original receipt records. The calibration of the balance will be observed and the techniques for pouring, weighing, measuring, and documenting the buffer preparation will be observed.
4 Interference 5 Check Sample 6 (ICS) Analysis	Not applicable
7 Initial Calibration 8 Blank (ICB) 9 Analysis	Not applicable
10 Initial Calibration 11 Verification 12 (ICV) Analysis	The laboratory technical personnel will be responsible for performing an independent check of the pH meter calibration once the pH meter has been calibrated using a pH 7 buffer and a buffer within 2 pH units of the expected pH of the samples. A third pH buffer that is 3 pH units lower than the second pH buffer will be measured. The calibration check will be documented.
13 Continuing 14 Calibration 15 Verification 16 (CCV) Analysis	The laboratory technical personnel is responsible for performing a continuing calibration check of the pH meter after every 10 th measurement. The pH buffer closest to the sample pH will be measured to verify that it is ± 0.05 pH units from the temperature-adjusted nominal value. The results of the CCVs will be documented in the instrument log.
17 Continuing 18 Calibration Blank 19 (CCB) Analysis	Not applicable
20 Spike Sample 21 Analysis	Not applicable
22 Calibration 23 Corrective Action 24 Audit	The QA Auditor will be responsible for auditing the calibration corrective action records. This will be accomplished by reviewing the calibration logs for deviations and reviewing the documented corrective action to ensure that both the immediate and long-term corrective actions are documented and implemented.
25 Method Blank 26 Analysis	Not applicable
27 Laboratory 28 Control Sample 29 (LCS) Analysis	Not applicable

**Table C-6. Examples of How Data Correctness Procedures Can Be Applied
Within Case Study C.2**

Procedure or Verification Step	Approach Taken in Case Study C.2
Instrument Inspection and Maintenance Audit	The QA Auditor will be responsible for conducting an audit of the instrument inspection and maintenance activities. This audit will be accomplished by reviewing the SOP requirements, the instrument logs, and the personnel training records. The documentation in the logs will be reviewed to ensure that the instruments are inspected and maintained according to the SOPs. Results of the audit will be documented in an audit report to the laboratory manager.
Instrument Calibration Review	The laboratory technical personnel will be responsible for performing an instrument calibration review. This review will be conducted at the end of each day to verify that calibration records have been completed in the instrument logs for all instruments used during each day of testing.
Data Recording Audit	The QA Auditor will be responsible for conducting a data recording audit. This audit will consist of a review of the raw data to ensure that raw data entries are in compliance with the data recording SOP: data are recorded in ink, any changes to data are documented so as not to obscure the original entry, which is crossed out with a single line, dated, initialed, and justified.
Data Reduction Audit	See Data Handling Verification.
Data Transformation Audit	Not applicable
Raw Data Audit	The QA Auditor will be responsible for performing the raw data review. In addition to the activities described in Data Handling Verification, this audit will include a "reasonableness" review to look for values that appear to be outliers or anomalies.

**Table C-7. Examples of How Data Consistency Procedures Can Be Applied
Within Case Study C.2**

Procedure or Verification Step	Approach Taken in Case Study C.2
Data Handling Audit	The laboratory technical personnel and QA Auditor will be responsible for data handling verification. The laboratory technical personnel will be responsible for checking 100% of hand-transcribed data or hand-calculated data. The QA Auditor will be responsible for recalculating selected test results and water quality data to verify that the reported data are correctly transcribed or calculated from the original daily observation sheets. 100% of data transcriptions to the probit program will be verified.
Data Transmittal Review	See Data Handling Verification.

**Table C-8. Examples of How Data Completeness Procedures Can Be Applied
Within Case Study C.2**

Procedure or Verification Step	Approach Taken in Case Study C.2
Documentation of Field Corrective Action	Not applicable
Sample Records Documentation and Audit	The POTW operator will be responsible for generating the COC forms. The Sample Custodian will be responsible for documenting sample receipt and for relinquishing the sample to the laboratory. The analyst will be responsible for daily records of sample handling and storage between test renewals during the effluent test. The COC form will prompt the custodian for all pertinent information related to sample receipt and condition. Completion of the COC form will include documentation that the sample was relinquished to the analyst. Daily storage and handling will be documented on a daily tracking form maintained by the analyst. When residual sample are returned to the custodian at the end of the test, a receipt and final disposition will be recorded on the COC form.
Sample Shipment Documentation and Audit	The POTW operator will be responsible for initial sample custody and handling, including choosing an appropriate transporter. The sample custodian will be responsible for initial lab custody, for maintaining the shipping papers, and for documenting the sample condition upon receipt. The QA Auditor will be responsible for reviewing the COC and handling records. Sample transfers (custody) and handling between the POTW and the laboratory will be documented using the COC form, the shipping receipts, and the sample receipt log.
Data Management Audit	The QA Auditor will be responsible for assessing the adequacy of data management practices. This will involve a walk-through of the entire project by tracing the sample via the paper trail from sample receipt through testing, to reporting and archival. Any required activity that is not supported by documentation or any gap in the sample testing process will be considered a failure of the data management system.
Chain of Custody Documentation	The laboratory sample custodian will be responsible sample custody. Documentation will consist of the sample custody form and a sample receipt form. The custodian will document any discrepancies and results of this communication on the COC form.
Sample Log In	The sample custodian will responsible for sample log in. Samples will be assigned a unique laboratory ID that will be documented on the COC form and the sample container. The sample condition (temperature, integrity) will be documented on the sample receipt form.
Sample Identification Audit	The QA Auditor will be responsible for performing the sample ID audit. The COC, sample container, and sample receipt log will be reviewed to verify that a unique ID is assigned to the sample and that the ID is documented on the sample container and the COC.
Instrument Inspection and Maintenance Documentation	The laboratory technical personnel will responsible for the inspection and maintenance of instruments. SOPs will define the precise items to be inspected, routine maintenance activities, and frequency. Daily inspection and maintenance will be performed as part of instrument calibration. Other maintenance (monthly) will be assigned to specific lab staff and posted. For example, for the DO meter this will involve checking the membrane for rips and air bubbles. For the pH meter this will involve checking the electrolyte level in the probe. The results of these activities will be documented in instrument-specific logbooks.

**Table C-8. Examples of How Data Completeness Procedures Can Be Applied
Within Case Study C.2**

Procedure or Verification Step	Approach Taken in Case Study C.2
Traceability of Standards Review	The laboratory technical personnel will be responsible for maintaining documentation that ensures traceability of analytical standards. All standards will be logged into the Laboratory Standard Receipt Log and assigned a unique ID that will be written on the standard label. The Receipt Log form will prompt for a complete history of the standard. The certificate of analysis received with standards will also be labeled with this ID and filed in a designated log book. The ID will be referenced in the calibration logs (e.g., for pH buffers).
Documentation of Calibration Corrective Action	The laboratory technical personnel will be responsible for documenting corrective action for failed calibrations or checks.
Laboratory Analysis Records Review	The laboratory technical personnel will be responsible for maintaining and reviewing all records related to the toxicity test. A standard project file “checklist” will be created to identify all test requirements and the location of supporting records. The full documentation package will be reviewed vs. the QAPP and SOPs to ensure that the test method requirements are met.
Documentation of QC Results including Control Charts	The laboratory technical personnel will be responsible for documenting the results of test quality control (the control survival) and the reference tests, including preparation and maintenance of the reference test control charts. The Laboratory Manager will be responsible for reviewing and verifying that the data are acceptable and traceable. The control survival results will be calculated and reported as part of the project files. The reference test will be run monthly, and all records will be maintained in a reference test log book. Each successive LC50 will be documented on the control chart.
Documentation of Laboratory Corrective Action	The laboratory technical personnel will be responsible for documenting corrective action for violations of general laboratory or testing procedures. Immediate corrective action will be documented directly in the instrument log or project files. Long-term corrective action will be addressed as changes to SOPs, forms, or re-training. These activities will be documented by the laboratory manager in response to QA audits or in memos that directed procedural changes.

Data Validation Within Case Study C.2

The validation process for Case Study C.2 is accomplished by reviewing the toxicity test conditions and results against the requirements of the POTW permit and the method. The validation process for toxicity testing considers the overall “success” of the test and weighs the relative impact of deviations on the final LC50.

The most critical elements of the toxicity test are those that would indicate that the testing system is out of control and therefore cause the test to be rejected. These elements include the survival of mysids in the control treatment and the results of the reference toxicant test.

Once the testing system is considered to be in control, the required test elements that can bias the test results are reviewed. These elements include the sample holding time and conditions, the age of the test animals, the acclimation of animals prior to testing, the water quality and

1 lighting conditions during the test, and the number of treatments and animals tested. Minor
2 excursions from the testing requirements for any one of these elements will not be likely to affect
3 the final LC50 value. However, multiple or extreme excursions can reduce or enhance short-term
4 mysid survival and thus alter the final test result. In all cases where testing requirements are
5 specifically or implicitly violated, then the data reviewer must weigh the number and type of
6 violations to determine whether the test results are still valid. For example, if the age of the test
7 animals is within 28 hours rather than 24, all other testing conditions are met, and the LC50 is
8 well below the permit limit, then the validator would probably consider the test valid. On the
9 other hand, if the age of the test animals is 28 hours, the water quality is erratic, and the LC50 is
10 within 10% of the permit limit, the test would likely be rejected. The technical experience of the
11 validator weighs heavily in these decisions.

12 Finally, the data reviewer must consider the supporting documentation. Major
13 documentation issues (the inability to confirm the test species, missing calibration records),
14 particularly representing a pattern of neglect, can be sufficient to fail a test.

1 **APPENDIX D**

2 **ISSUES CONCERNING THE VALIDATION OF**
3 **DATA VERIFICATION SOFTWARE**

4 Elements that are typical of software and hardware verification and validation procedures
5 that would have been performed are included below for added clarification and information. This
6 information can be found in the following guide:

7 *Guide To Inspection of Computerized Systems in Drug Processing.* Division of
8 Drug Quality Compliance (HFN-320), Associate Director for Compliance, Office
9 of Drugs, National Center for Drugs and Biologics, and Division of Field
10 Investigations (HFO-500), 1983.

11 Simulation should be performed to ensure that the software will consistently perform the
12 expected task within operational limits. The individual verifying the software and the methods
13 used must be reported. Software verification should include the following actions:

- 14 • Determining if the program matches the operational functions (i.e., does the
15 software generate records, analytical instrument outputs, monitoring equipment).
- 16 • Determining whether the conditions for the worst-case scenario have been tested,
17 (i.e., process speed, data volume and frequency). Data field size should be
18 considered, and the maximum number of characters should be considered for
19 verification.
- 20 • Determining whether replicate measurements were used to determine program
21 bugs and assure consistent results.
- 22 • Verifying that proper documentation is recorded and that the names of individuals
23 or vendors involved in the verification are included in the documentation. The
24 verified protocol and all pertinent information should be fully documented.
- 25 • Determining whether the data verification protocol includes a system that allows
26 changes to be made and verification to be redone easily.